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THE INSTITUTE OF PETROLEUM

AN Ordinary General Meeting of the Institute was held at 61 New Cavendish Street, London, W.1, on 3 November 1958, the Chair being taken by W. S. Ault, a vice-president of the Institute.

The General Secretary read the minutes of the previous meeting, which were confirmed and signed as a correct record. He also announced the names of members elected since the previous meeting.

The following paper was then presented in summary.

SOME PROBLEMS OF PETROLEUM GEOLOGY IN KUWAIT*

By A. F. FOX†

SUMMARY

The Geological succession in Kuwait is related to the successions in Al Hasa and in S. Iraq and points of doubtful correlation are noted. Tectonic history and Palaeogeography are briefly reviewed. The origins of the Burgan, Magwa, and Ahmadi structures are examined in detail, a combination of salt intrusion and folding being suggested as a possible cause. The distribution of the crude oil in the three reservoirs is described and selective absorption during migration through sandstone suggested as an explanation.

INTRODUCTION

THE position of Kuwait is unique. This small country, only about 6000 square miles in area, is the geographical and geological hinge pin of the Persian Gulf geosyncline. It stands at the edge of the Arabian foreland, at the limit of the Tigris–Euphrates sedimentary basin and at the limit of the Zagros orogeny. Between its boundaries the geological formations alter from the Iraq type on the N. to the Saudi Arabian on the S. and contain in them what is perhaps the largest existing single body of crude oil.

The geology of Kuwait is therefore of special interest to all members of the IP, but as it has already been adequately described,[1, 2] in this paper reference to the geological background will be made only in order to introduce some new information which has become available from the surrounding countries since the previous papers were written. Here some of the unsolved problems with which we are at present concerned are presented in the hope that the statement of the problems with an account of the evidence available may stimulate further research or may bring to light additional facts which will help towards their solution.

STRATIGRAPHY

At the 20th session of the International Geological Congress several papers on Middle East geology were published,[2–5] and much more information became available about the characteristics of the various formations at their outcrops, in Saudi Arabia on the one side and in Iraq and Persia on the other. For the first time it was possible to relate the Kuwait Succession, shown in detail in Fig 1, to the geology to the N. and to the S. A suggested relationship between sections in S. Iraq, Kuwait, and Al Hasa is shown in Fig 2. This correlation is drawn almost entirely on a lithological basis, but it is thought that the facies lines approximate to time lines on this N. to S. section, which is approximately parallel to the most likely direction of the shore line.

The total thickness of sediments in Kuwait is not known. Calculations from geophysical observations indicate that the basement is at least 20,000 ft below the surface. So far 13,853 ft have been penetrated at Burgan and, at that depth, the rocks were believed to be Triassic in age. The Saudi Arabian outcrop section shows about 2000 metres or 6500 ft below the equivalent horizon, while the upper part of the outcrop section is only about half as thick as the equivalent beds in the centre of the basin. It would therefore be reasonable to expect about 13,500 ft below the Trias in Kuwait, making a total sedimentary column about 27,000 ft, in reasonable agreement with the geophysical results. A thickness of about 25,000 ft is therefore a fair estimate for the total thickness of the sedimentary column.

The earliest deposits were probably laid down in the Redlichia Sea, though the Cambrian sandstones might have been terrestrial deposits. Later rocks were also laid down in the geosynclinal forming the successor to the Redlichia Sea and the deepest part of which

* MS received 25 August 1958.

† Kuwait Oil Co. Ltd.

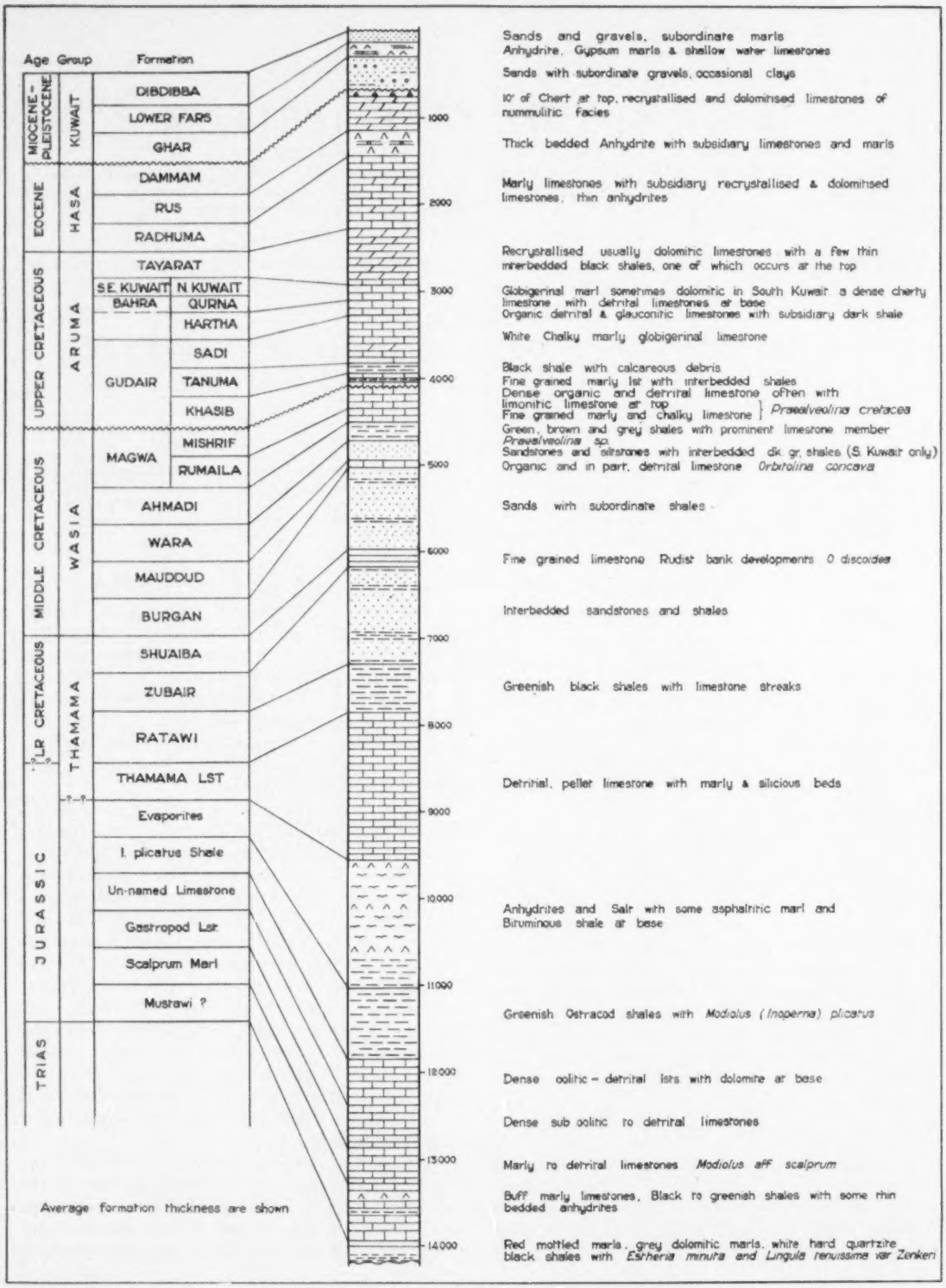

FIG 1

GENERALIZED GEOLOGICAL SUCCESSION IN KUWAIT

approximated to the present centre of the Persian Gulf.

In the Devonian, chalky and dense limestones, gypsiferous clays, shales, and marls are recorded at outcrop with some red shales in the Permian, indicating shallow and fluctuating waters in a near shore location. In coastal Hasa the Permian (Kauff) is stated to be a limestone of shallow water origin with some intercalated anhydrite beds indicating continuing shallow water conditions, probably isolated from land-derived material and with lagoonal phases.

The Trias is known in Kuwait and is represented there by black shales, quarzitic sandstone, grey dolomitic and red mottled marls. In Hasa it is composed of limestone, anhydrite, and red and green shales at the base, with a middle dolomite–anhydrite series and an upper shale–sandstone unit. At outcrop it is predominantly sandstone with minor red and green shales and impure limestones. The sea, therefore, continued in existence throughout the Trias.

The Jurassic throughout the area is composed of limestones with an evaporite series (Hith and Arab formations) at the top. At outcrop shallow water pellet limestones are found, and these occur sporadically in the coastal sections. Shallow waters were therefore common during much of the Jurassic and, at the end of this period, vast lagoons must have been present in which the evaporites were deposited. In Kuwait the evaporite series includes considerable thicknesses of bedded salt in one minor and three

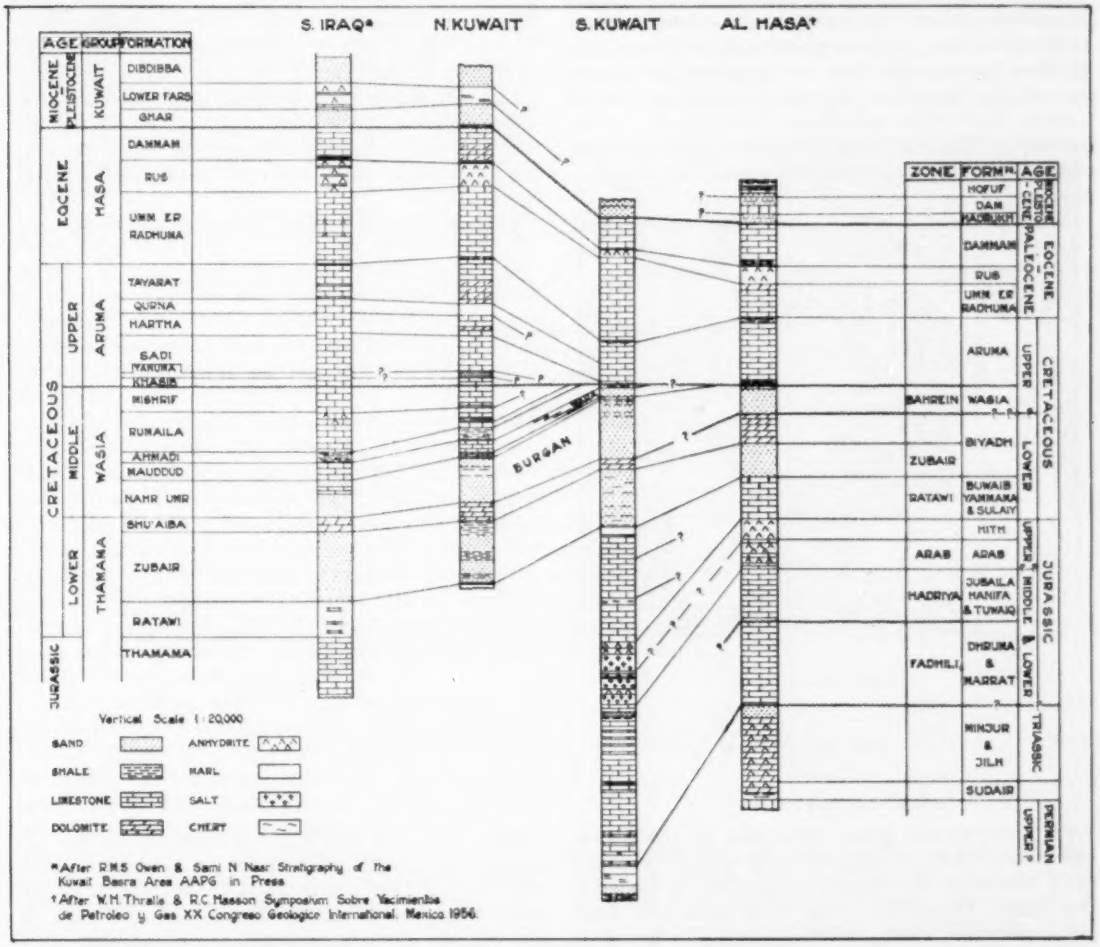

FIG 2

GEOLOGICAL CORRELATION FROM S. IRAQ THROUGH KUWAIT TO AL HASA

major cycles of evaporite deposition, but to the S. only anhydrites and limestones are recorded in the interval. Evaporite deposits of this type are characteristic of lagoonal conditions, and it is possible that the W. shore of the Zagros Geosynclinal Sea consisted during Jurassic times of a vast series of lagoons separated from the main ocean by a barrier reef. In normal evaporite deposits (if any such exist) the order of deposition should be calcium carbonate–calcium sulphate–sodium chloride–magnesium sulphate. In Al Hasa the sequence does not appear to have gone beyond the deposition of calcium sulphate, but in Kuwait there must have been several breaks of the barrier by the sea, which refreshed the contained waters, enabling them to sustain another depositional sequence.

The Upper Jurassic evaporites passed into shallow water detrital and pellet limestones in the Lower Cretaceous, which in turn gave place to the first of the great sandstone oil reservoir formations of the area, the Zubair Formation. This thick sand and shale series, normally about 1000 ft thick, can be traced as far south as Al Hasa, where it is productive at Ras Safaniya [6] and as far north as Zubair in S. Iraq, and possibly as far as Awasil, near Hit.[7]

Overlying the Zubair is the Shua'iba Limestone, a dolomitic and heavily fractured limestone from which oil has not yet been produced, and is immediately followed by the second sand reservoir formation, the Burgan. Here again sandy and shaly beds reach thicknesses of about 1000 ft and can be traced laterally from Dukhan in the Qatar peninsula to Awasil in Iraq, and may extend round the northern nose of the Arabian Shield into Palestine, where sandstones, shales, and clays occur beneath a limestone bearing *Orbitolina concava*, which is characteristic of the Mauddud Limestone in the Persian Gulf.[8]

In Kuwait both the clastic formations are thought to be derived from the Arabian massif, as there is a definite gradation in grain size in the Burgan Formation with the coarser deposits towards the west.

Above the Madudud Limestone in S. Kuwait, and also in the Neutral Zone, there is a local sandy development recognized as the Wara Formation, but from the upper part of the Middle Cretaceous a long succession ensued during which shallow water limestones and shales were deposited until Eocene times, which ended with a terrestrial phase. Sandy deposition returned in the Miocene and has continued into the Pleistocene with recent sub-aerial and alluvial deposits.

There are several minor discontinuities in the succession, but only two major unconformities are detectable in Kuwait. The main one at the junction between the Aruma and Wasia groups affects all the surrounding areas. Over structural highs such as the Burgan uplift, all the Magwa Formation and a great part of the Ahmadi Formation is missing and, although the deposition was attentuated against the rising structures, much of the loss must have been due to erosion. Another unconformity occurs at the top of the Dammam Limestone and marks a land phase during the Oligocene.

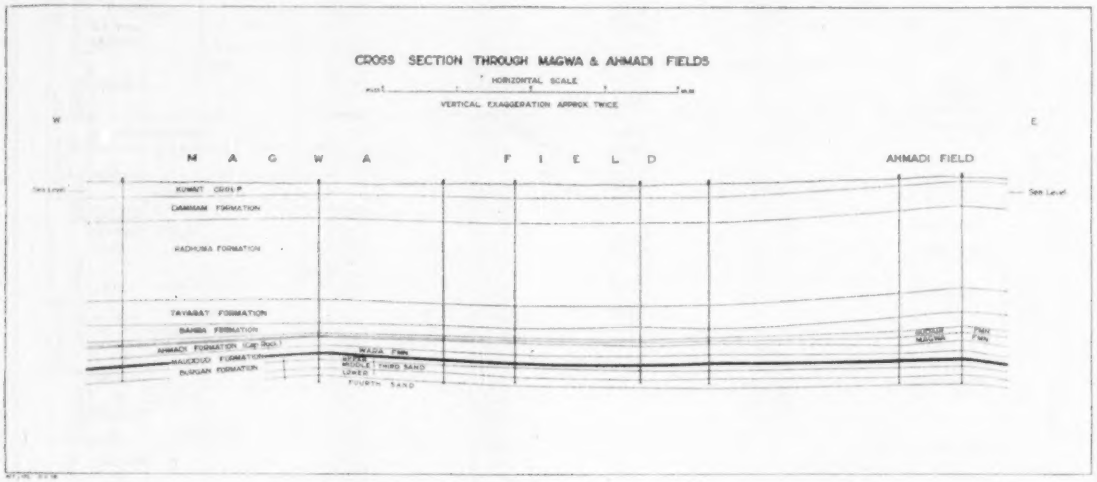

FIG 3

TECTONICS

The tectonic history of the area is relatively simple. The permanence of the Redlichia-Tethys Sea is demonstrated by the continuance of sedimentation, and steady downwarping to accommodate the volume of sediments must have occurred from Cambrian to

Middle Cretaceous times. The trend was reversed temporarily and evidence of uplift and erosion appears at the Aruma–Wasia unconformity. General subsidence continued during the Upper Cretaceous and the Eocene, although the main structures continued rising relative to their surroundings. At the end of the Eocene a period of uplift resulted in the surface of the Dammam Limestone being peneplained and slightly dissected. During the Zagros orogeny the force which caused the violent overthrusts in Persia was felt in a much diminished scale in Kuwait, where its expression is limited to gentle folds of the " plains " type. Downwarping is still taking place at the head of the Persian Gulf, and has probably been going on continuously since prehistoric times.

These post-Eocene folds are superimposed in Kuwait upon the earlier uplifts, which had been steadily increasing in size at least since the Upper Cretaceous and which may have been in existence even earlier. It is not easy to separate the effects of the uplifts from the results of Zagros foreland folding, but in SE. Kuwait the difference between the two types of structure is thought to be shown in two ways.

In the first place the date of uplift can be demonstrated by the relative thicknesses of the pre- and post-Wasia beds. This is best illustrated by a cross-section from E. to W. across the Magwa and Ahmadi fields (Fig 3), where it can be seen that over the crest of the Magwa structure (which was early in origin but continued to grow steadily afterwards) there has been a considerable thinning of the Magwa formation. This has not occurred at Ahmadi, where there can have been little uplift before the Eocene and very little before the Aruma–Wasia unconformity.

The types of structure can also be separated. On the one hand, the Burgan type structure with its oval outline and radial fault pattern is typical of uplift structures. On the other hand, the elongated anticlinal folds of the Ahmadi type are typical of the foothill folds of the Zagros mountain foothill belt and of compressive folding. At Ahmadi the intensity of the folding cannot be compared to that found in Persia, but the direction of its long axis is similar, while the fault pattern seems to run where the major strain axes would lie if the main thrust had been felt from Persia, although since it is difficult to trace faults from well to well, the true fault pattern may be rather different from the supposed one.

PALÆOGEOGRAPHY

In general, the distribution of land and sea was as shown on the map in Fig 4, which represents conditions in Callovian–Oxfordian times. Both later and earlier than this era the northern shores of the Zagros Geosynclinal Sea moved farther N. and E. The Arabian shore line must have remained more or less where shown, although the depths of water in the near shore zone fluctuated between fairly wide limits, as indicated by the variations in the types of deposit.

THE ORIGIN OF THE BURGAN–MAGWA–AHMADI STRUCTURES

The forms of the Burgan, Magwa, and Ahmadi structures have already been mentioned as indicating a later origin for the Ahmadi fold. The basic problem is, however, the reason for the formation of the Burgan and Magwa features. The fault pattern on these structures and the oval shape of the contour lines

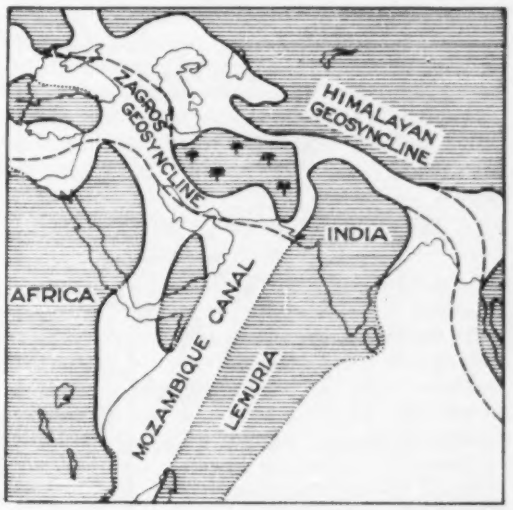

FIG 4

JURASSIC PALÆOGEOGRAPHY

suggest that they are both uplifts. It might be suggested that their formation was due to differential compaction, an opinion which seems most attractive in view of the large volumes of sand and shale in the section and the inference of a large buried structure which might be drawn from the effect of the unconformity shown in Fig 2. But if differential compaction had been the cause it would have been expected to have been consistent in operation. If flank wells on the two structures are examined, it is seen that Burgan received a greater proportion of deposits in the Maestrichtian than did Magwa, indicating that the Burgan was relatively lower during Cenomanian times but that the discrepancy was evened out during or just prior to the Maestrichtian. It is difficult to see how such a difference could have been caused by differential compaction. One possibility is that when deposition takes place over a series of hills on an old

erosion surface the type of deposit is dependent upon the position around the hill. Differences in the shape of the primary feature might therefore cause characteristic depositional anomalies, which after compaction might lead to differences in relative elevations.

The uplift hypothesis is, however, at the moment more attractive, especially since laboratory experiments investigating this mechanism of rock deformation have succeeded in reproducing closely the actual

After C.A.E. O'BRIEN

FIG 5

DISTRIBUTION OF SALT PLUGS IN THE MIDDLE EAST

fault pattern.[9] What then caused the uplift? Only two mechanisms could reasonably explain the result, either an igneous or a salt intrusion. Igneous intrusion is always possible, but as a complete explanation raises more difficulties than are reasonable. It would first be necessary to imagine sporadic igneous activity in an area where igneous rocks are practically unknown later than the Cambrian. Secondly, this igneous intrusion would have to continue at intervals over very long periods of time (at least from the Lower Cretaceous to the Eocene). Thirdly, its expression would have to have been limited to a relatively narrow belt; and fourthly, the expression of igneous activity must be limited to deep seated intrusion alone.

Salt offers by far the simplest explanation and the results of salt movement can be seen in the surrounding countries. The nearest surface expression which shows the characteristics of a piercement salt plug, although no salt actually emerges, is the Jebel Sanam, SW. of the Zubair oilfield and NW. from the field at Raudhatain, but Fig 5 shows how widespread are the salt intrusions in the area, particularly those of Cambrian age.

There has never been a satisfactory and complete explanation for several of the phenomena associated with salt intrusives, but O'Brien[10] has recently suggested an elegant explanation for many of the hitherto seemingly contradictory occurrences. In brief, O'Brien, while retaining density difference as the motive force causing salt movement, relates the occurrence of an intrusive salt plug in S. Persia to four stages. The first consists of the intrusion of igneous necks into an overlying sedimentary series containing bedded salt. The second covers rapid crystallization of the igneous rock in the highly thermally conductive salt bed, with a reduction of volume on crystallization. In this stage too the salt infiltrates round the plug into the area of low pressure formed at the contact of the intrusive with the country rocks when the intrusive contracts on freezing. At stage three a further flow of salt occurs round the igneous plug according to the conditions set up by the weight of the overlying sediments. The start of this stage is dependent on there being a sufficient overburden to provide the density difference necessary for salt movement. Its onset might be delayed indefinitely after stage 2, and the depth of overburden is always critical for its continuance. During this stage erratic fragments may be plucked off the intrusive plug by the salt flowing past it. At various times the salt plug will completely penetrate the overlying sediments, and this penetration is stage 4. In this sequence an intrusive salt plug at Burgan must be imagined to be in stage 3, while the preliminary igneous activity could have been limited to a short period sometime after the Cambrian.

The total uplift of the Burgan structure must have been more than 1300 ft, and it has been going on continuously but rather erratically since the early Cretaceous or before.

THE ORIGIN OF THE AHMADI RIDGE STRUCTURE

The Arabian foreland[11] has not been exposed to tangential stress between Cambrian and Eocene times so far as can be determined. Before the Zagros

orogeny had occurred, therefore, the vast sheets of sediments would have shown any disturbance only where they had been uplifted by intrusions, where the basement had so great a relief as to affect some 20,000 ft of sedimentary cover, where there had been basement subsidence, or where movement on old fault planes had continued.

Where gentle uplifts had occurred, as at Burgan, the sedimentary rocks, the majority of which are limestones, had undergone a considerable deformation, the strains due to which were very small because of the long time over which the movement was spread. It is suggested that this gentle deformation might cause an increase in the mechanical strength of the beds affected by it in the same way as the strength of a piece of metal can be increased by the process known as " work hardening." The writer has not been able to find any direct supporting evidence for this supposition, the available accounts of experimental work on rock strengths so far not having produced results in a suitable form. It will be interesting to see whether further research reveals a similarity in the behaviour of rocks and metals in this instance, as well as in the more spectacular effects of the sudden release of stress which causes both metals and rocks to shatter.

If it were correct, a compressive stress, such as that due to a distant orogeny affecting a large area of sediments, would give rise to areas of maximum strain which would occur at positions where Young's modulus of the beds was higher than normal. The Burgan–Magwa structure would therefore have to be regarded as a " toughened " block, for which the stress due to the Zagros compression was below the elastic limit; the limit would, however, be exceeded in the immediately adjacent beds, which would be deformed by folding, in this case into an anticline. As the age of the Ahmadi fold corresponds with that of the Zagros orogeny and the fault pattern on the Ahmadi structure, so far as it can be determined, appears to correspond more nearly with one due to a NE.–SW. compression than to one caused by uplift, the fault pattern has been used as supporting evidence for the force being a NE.–SW. compressive one.

There is, however, a major objection to this which so far has not been explained. The majority of faulting on the Burgan, Magwa, and Ahmadi structures is pre-Maestrichtian, and only in the case of the main Graben feature can evidence of faulting be found in the higher beds. If the Ahmadi structure is due to post-Eocene forces it would be expected that a fault pattern set up at that time would be apparent in the Eocene rocks. It is in fact impossible to state definitely that the Dammam Limestone is not faulted over the Ahmadi Ridge structure, but drilling evidence is sufficient to show that any faults which do exist in beds above the Aruma–Wasia unconformity must have very small throws, while those occurring below the unconformity have larger throws and must have been in movement in pre-Maestrichtian times.

Considering only the increases in formation thicknesses away from the Magwa crest, which is illustrated in the cross-section (Fig 3), it must be concluded that the Ahmadi Ridge fold is probably post-Eocene in age.

The faulting cannot have been caused by a movement later than the Aruma–Wasia unconformity, or it would extend into the beds overlying the Magwa Formation, which it apparently does not do. The faulting, therefore, cannot be associated with the folding and must have been caused either by an earlier compressive stress acting NE.–SW., by uplift, or by differential compaction. Which of these has in fact caused the fractures is not known. Perhaps further drilling will enable the faults to be located more accurately and their throws and vertical extents determined; until that is possible the problem must remain unsolved.

THE ORIGIN AND MIGRATION OF THE OIL

The accumulation of such a vast quantity of oil as is present in the oilfields of SE. Kuwait is a problem which must be of interest to everyone concerned with the oil industry. There are protagonists of both the main theories of oil pool formation, one school insisting that all crude migrates long distances into every reservoir, and the other insisting on local formation and accumulation. Before discussing the relative merits of the two as explanations of the SE. Kuwait accumulations, it would be as well to give a summary of relevant data.

The structure of the Burgan–Magwa–Ahmadi reservoir is composite; it can be regarded as a common reservoir of very large area but relatively small depth, upon which are superimposed three subsidiary reservoir structures isolated from each other. So far as it has been possible to tell, the oil in the basal, common part of the reservoir is of a similar composition at any one level in it. There is, however, a steady increase in specific gravity with depth. The crudes in each of the three subsidiary reservoirs are, however, slightly different in composition. That in the Ahmadi structure is lightest, that in Magwa intermediate, and the Burgan structure contains the heaviest oil. An additional point may be added here that the Wafra field in the Neutral Zone to the S. of Burgan appears to produce crude of a heavier specific gravity than Burgan. Furthermore, there was no original free gas at Burgan; a very small quantity was present in Magwa and an appreciable amount in Ahmadi.

The sedimentary basin in which the oil must have been formed is the Zagros geosynclinal, the centre of Cretaceous deposition seeming to lie to the NE. of the position of the Raudhatain oilfield. The sandy formations which are oil-bearing in Kuwait probably

give way westwards to coarser and coarser near-shore deposits until they vanish at the Cretaceous shore line. Eastwards they probably give place to deeper water deposits which form the poly-bituminous marls and marly limestones and the massive limestones of the Persian foothills.[12]

Trask [13] obtained up to three gallons of crude petroleum per ton from recent sediments by distillation. If it is supposed that natural processes would give a comparable yield from ancient sediments, the crude originally in Burgan, Magwa, and Ahmadi could have been formed in the Burgan formation alone within a radius of a few tens of miles. If the shales alone contributed to the generation of oil, the radius would be increased about twice, but if lower formations contributed also, the radius could be reduced to quite small dimensions. In a private communication, B. Bonnett has informed the writer that recent work carried out at Imperial College seems to indicate that part at least of the crude oil in the sands of the Wara formation on the Burgan field may be derived from the immediately adjacent shales in the same formation.

There exist throughout the reservoirs in SE. Kuwait pockets of heavy oil or tar surrounded by lighter crude. It has been supposed that these might be the vestiges of an earlier generation of crude oil, the lighter proportions of which had been lost by seepage either along fault planes or other free channels.[14] It is, however, equally possible that these local heavy oil patches have remained where they were actually formed and have been surrounded by crude which was altered by its passage through the formation. This explanation is to some extent favoured by the fact that the patches of heavy oil are found both above and below the oil–water contact and even at some distance away from the reservoir.

It has already been stated that the properties of the crude oil in the SE. Kuwait reservoirs vary with depth. This variation is symmetrical, *i.e.* a sample of crude obtained from a certain depth range on one side of the field will be identical with a sample obtained from a similar depth range anywhere else except if the sampling points happen to lie on the three isolated high spots where the crude is symmetrically distributed in each high. This " radial " variation in quality could be explained if the crude had flowed into the reservoir equally from all directions.

It seems, from the information at present available to us, unlikely that crude oil was within the actual boundaries of the oil pools in which it is found, unless the quantity of crude obtained from ancient sediments was markedly greater than that obtained from recent ones. Migration of crude over some distance is always necessary even if formation was local to the reservoir, the probable minimum distance being of the order of tens of miles mentioned above. However, if migration over distances of this order is inescapable, it follows that migration over longer distances must also be possible, granted only a longer time for it to take place. It is also generally accepted that the most likely source rocks for petroleum generation are those organic shales and limestones abnormally rich in bituminous matter. If this is correct the oil now filling the oilfields of SE. Kuwait must have travelled considerable distances from its place of formation, which was most probably the centre of the depositional basin.

There is also the distribution of the crude in the three high spots in the structural complex to be considered. At the crest of the Ahmadi structure free gas was originally present, and the crude immediately beneath was the higher in API gravity. At Magwa there was probably a very little free gas and the crude was slightly heavier than that at Ahmadi. At Burgan there was no free gas and the crude was the heaviest of the three. It has been suggested that the gas distribution in structures in rising sequence on a structural trend can be explained by selective entrapment.[15] It is difficult to see how such a distribution would result if the generation of the crude had been local and migration radial into the reservoir.

The distribution of the crude in the reservoir, if the differences in the subsidiary highs are ignored, has been variously explained. From a purely physical standpoint it has been shown that the earth's gravitational field should cause progressive decrease in the lighter constituents with depth.[16] From a geological point of view, it is very difficult to imagine that a body of homogeneous crude contained in the pores in a reservoir consisting, as does the Burgan Sand reservoir, of alternating sands and shales, could separate by movement even of molecules. Not only are the pore spaces in the shales across which movement would have to take place of molecular dimension, but in some cases the oil column is interrupted by beds which are water-saturated and contain no oil. Although these beds are probably discontinuous, they would provide barriers at which local concentrations of heavy molecules should be found if segregation had taken place because of the forces due to gravity. No evidence for such accumulations has been found in the SE. Kuwait fields. More especially, the cases where lithological barriers do not occur, *i.e.* fractured limestone reservoirs, should exhibit the same increase in heavy molecules towards the base of their oil columns. There are, it is true, few reservoirs with oil columns large enough for the effect to be noticeable; there are even fewer where testing has been such as to reveal the difference if it did exist. However, the Persian reservoir at Gatch Saran apparently showed no difference in density between crudes taken from the top and the bottom of an oil column of over 4000 ft.[17]

Fig 6 is a photograph of part of a core taken in the Wara formation in a Burgan well showing part of one of the pockets of heavy oil. It will be noted that in the photograph the darker heavy oil in the thin sands in the centre of the specimen appears to occupy the top part of the bed, the bottom portion being occupied by lighter oil, which appears to have moved into position after the tarry oil. The orientation of this specimen is not certain, however. No gravity segregation is apparent. The lighter coloured bands are dense, water-saturated siltstones which would apparently form a barrier even to molecular movement of crude oil.

On the other hand, it is known that certain sands,[18] and also fuller's earth,[19] have the property of preferentially adsorbing the blackest and heaviest constituents from a crude oil passing through them. The oil adsorbed can be removed by water flushing by a brine. A body of crude oil migrating through a sandstone could therefore be expected to have its heavy molecules adsorbed and held until the crude had passed, when they would be flushed off by formation water. Some of the isolated heavy oil patches in SE. Kuwait could be the remains of such an occurrence.

With this argument it can be imagined that crude oil forms in all sediments but only from the rich " source " sediments is sufficient produced for migration to be possible. The crude then travels through the rocks and when these are sandstones is subjected to a filtering action in which the heavy molecules are adsorbed on the surfaces of activated quartz grains. This filtration alters the character of the body of crude from an homogeneous one to a body with light crude in the van, followed by increasingly heavy liquid. The last crude will be the heaviest and darkest fraction capable of movement.

Reservoirs will be filled in order from the structurally lowest to the highest with successively heavier crude. The lowest reservoir will therefore receive the lightest crude and the highest reservoir the heaviest. Gas distribution will be on the same basis following Gussow's hypothesis. The crude in each reservoir will be graded in depth, and the quality of crude will vary between separate reservoirs.

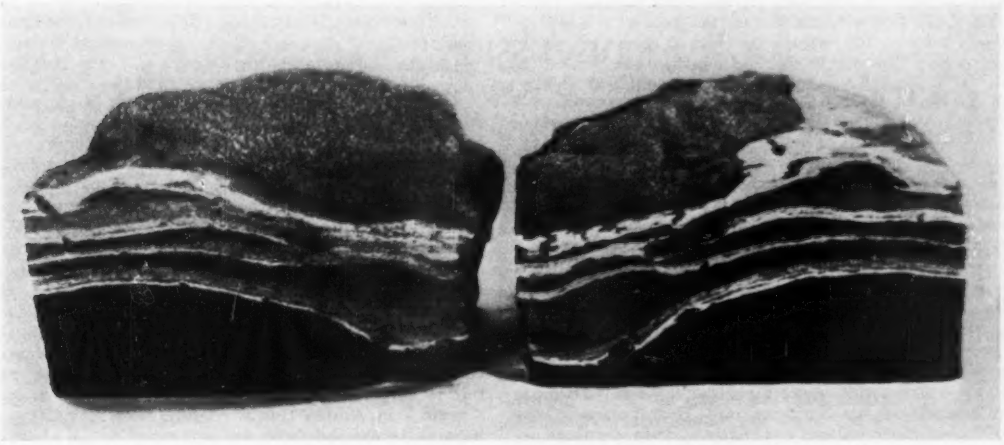

Fig 6

CORE SPECIMEN FROM A BURGAN WELL

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DISCUSSION

Dr F. R. S. Henson: This paper is welcomed as a valuable new contribution by the author to the literature of the world's largest oilfield.

There is little ground for comment on the stratigraphical sections, except perhaps to qualify the classification of the productive "Arab zone" as a lagoonal deposit. The use of this term may be misleading.

Excluding Kuwait, subdivisions of the "Arab zone" are remarkably constant over long distances. A wide, level floor of deposition may be imagined, rather than a series of lagoons.

A reef barrier has been assumed to account for high sea-water salinity.

Small, lenticular stromatopore reefs do occur in the Arab zone limestones, and a marginal calcarenite bar has been postulated by Steineke and Bramkamp in the "D" limestone member; but the anhydrite members are very constant until they tongue out into normal marine limestones in Trucial Oman.

This transition, without any signs of barriers or stratigraphical breaks, is significant because it indicates a passage from normal marine to evaporite facies in a continuous area of deposition which is described by Steineke and Bramkamp as the "Hasa structured terrae."

The lithology and fauna of the Arab zone limestones point on the whole to a shallow-water environment, and it is suggested that here, as suggested by R. M. Ramsden in an unpublished thesis, high salinity may have been associated with submarine plateau conditions where stagnation with rapid evaporation prevailed in the absence of deep-water current circulation.

These conditions may also have been favourable for indigenous oil formation.

The author's views on the tectonics of Kuwait are very interesting, including some new and stimulating ideas. He suggests different origins respectively for the Ahmadi structure and for the Burgan and Magwa structures.

The Ahmadi structure is attributed to compression from the Zagros orogen. However, I join with the late G. M. Lees in doubting the ability of relatively weak sedimentary layers to transmit tangential stresses over the relevant distance of 260 miles from the Zagros thrust front, especially as Kuwait is on the foreland slope of the Persian Gulf depression and few of the intervening folds are "closed."

The Kuwait uplift as a whole trends N.–S. and presumably belongs to the system of dominantly N.–S. folds extending from Qatar through the Hasa province of Saudi Arabia, so that its origin is probably related to that of the whole group.

The strike of these structures is in marked contrast to that of the nearest Zagros folds which trend NW.–SE. However, N.–S. structures appear occasionally in the Zagros fold-front, where anticlines with the normal NW.–SE. strike tend to pitch out along N.–S. cross-trends, as in the Bushire structural embayment, which lies significantly on a northward projection of the Qatar Peninsula strike. De Boeckh attributed these anomalies to interference effects of Palæozoic features at depth; but it is to be noted also that the ancient basement "grain" of Arabia trends mainly N.–S., and this may have had some influence through reactivation of old lines of weakness.

Recognition of this possibility may have prompted Thralls and Hasson to suggest that the greater Gawar anticline, a long N.–S. fold in Hasa, is due to "shearing in the basement."

These authors do not indicate how shearing in the basement might produce a parallel surface anticline where no superficial wrench-faulting is visible.

Quennell's analysis of ancillary shearing effects on a larger scale in the Dead Sea rift valley provides food for thought on this subject; but perhaps the most interesting comparative evidence is to be found in the Jabal Abd-el-Aziz structure in the North Syrian foreland, where vertical, diapiric upthrusting of deep rocks first produced a surface anticline and then burst through it.

A probable earlier stage in this process is seen in two neighbouring structures on the same trend, namely, the Jabal Sinjar and the Jabal Butmah, which reveal distension faulting, and in the latter case a buried graben, in the axial zones.

These features are not normal buckling folds and are believed to be associated with deep-seated shearing movements.

Thralls and Hasson's view of the Ghawar fold could perhaps be interpreted along these lines, and applied also to the N.–S. Kuwait uplift.

However, as the author suggests, the rising of salt plugs would have similar effects and might provide a better explanation for the more domal anticlines of Kuwait. But salt doming does not account for the

pronounced N.–S. Arabian trend of the Kuwait uplift as a whole, unless a salt ridge condition by deep structure is visualized.

As the author observes, the origin of salt domes is still obscure.

Differential loading may explain the progressive rise of a salt plug once a salt column has been established, but some other cause must be found for the initial, very localized, escape of salt through a roof which elsewhere successfully confines the salt source. Neither orogenic folding nor O'Brien's ingenious association of salt plugs with igneous extrusions can account for some newly-found Cambrian salt extrusions, devoid of igneous debris, far out in the unfolded Arabian foreland.

However, other possibilities arise with the discovery that important tangential movements of the basement occurred in the Oman foreland at or towards the end of the Cretaceous, probably with minor recurrences thereafter; the larger displacements are expressed in wrench-faulting at the surface, but there is evidence to suggest that small movements of the basement occurred without rupturing the overburden. In this process the Cambrian salt doubtless acted as a lubricant, suffering severe drag-stresses which would enable it to burst through its roof at weak points, for example, where cross-faulting cut trap-doors for its escape.

In these circumstances it would not be surprising to find salt domes aligned frequently, but by no means exclusively, along basement wrench-faults.

According to these speculations, local salt diapirism at Kuwait, and the squeezing up of weak, dissected material to produce the Arabian anticlines along buried N.–S. shear zones, could be attributed to the same tectonic region of basement block-jostling, stimulated by major tangential movements farther to the S. I would welcome the author's views on these topics.

The section of the paper dealing with the origin and migration of the Kuwait oil is extremely interesting, and presents a challenge for discussion by chemists and petroleum engineers.

One point on which chemical opinion would be useful is the possibility that oils originally of different composition may be variably mixed in the Kuwait reservoir.

The equivalent sands in the Basrah fields contain considerable quantities of land-plant debris and are generally considered to be estuarine. If any indigenous oil was formed in these beds it might differ in composition from that originating in normal marine sediments to the NE.

It is surprising that structures which have suffered considerable distension-faulting after deposition of their reservoir rocks should have retained so much oil.

Is it possible that the heavy oil pockets, to which the author refers, are residues of oil which escaped from deeper sources during the Pre-Maestrichtian distension phase? Or could they be residues of Cretaceous oil, most of which escaped at that time?

Continued peripheral subsidence would presumably extend the migration range for new accumulation.

Perhaps the author would care to comment on these points and on the large Burgan seepage, and its possible effects on the composition of the underlying reserves.

I would like to thank and congratulate the author for his thoughtful paper, which has given me much enjoyment.

A. F. Fox: It is true, as Dr Henson points out, that the Arab zone facies points to a shallow-water environment, and there is a transition to a normal shallow-water facies, farther to the S. I believe, however, that the analogy with the shallow-water banks in the Caribbean is not strictly true, at least in the N. of the Zagros geosyncline. If a section from S. Iraq, to the N. of Saudi Arabia at Al Jauf, is examined it will be seen that there appears to be a steady rise of the various structures superimposed on it at Zubair, Raudhatain, Magwa, Burgan, and Wafra, while at the Aruma/Wasia unconformity near the boundary between Saudi Arabia and the Kuwait/Saudi Arabia Neutral Zone there appears to have been a total erosion of the Burgan sands. It appears also that there is a structural fall southwards away from Al Jauf.

It is possible, therefore, that N. of the Gulf is separated from the southern part by a structurally positive area trending E. to W. It is interesting to note also that N. L. Falcon, in a recent paper,* has suggested that there may be a structurally positive area separating the Persian oilfields from the S. Iraq and Kuwait oilfields. The Cretaceous and Tertiary basin in which the Persian oilfields lie seems to tail out somewhere under Abadan, and the oilfields on the other side of the Gulf may belong to a separate depositional basin or sub-basin.

If the southern part of the geosyncline was separated from the northern part and if there was a structurally positive area dividing the northern part into two halves, these factors alone would have been sufficient to allow the formation of a lagoon, in which I have imagined the evaporite succession to have been deposited, on the western side of this geosyncline.

We know very little about transmission of stress through sediments, and certainly if they are horizontal, or even if they are domed upwards, it is difficult to imagine that any stress at all could be transmitted through sandstones or even limestones. In this case, however, the beds formed the concave lining of a synclinal basin, so that a tangential stress might be transmitted through them, as any downward buckling would be prevented by the underlying beds, the whole achieving a somewhat spurious rigidity.

If uprising salt provided the active power for the Burgan and Magwa uplifts, it is very probable that the position of the salt intrusions was controlled by a deep Palæozoic structure.

Dr Henson's suggestion of wrench faulting with salt intrusives is most interesting, as is his news of the discovery of Cambrian salt intrusives on the Arabian foreland. Certainly a great deal of disturbance could occur beneath a blanket of plastic salt beds, with but indirect effects on the beds above the salt. If igneous intrusion does initiate salt intrusives, then several igneous intrusions, arranged lineally, could give rise to a composite salt plug able to form a structure as elongated as Ghawa.

The problem of the oils in these reservoirs is extremely complicated, the more so because it is almost impossible to sample sufficiently finely to give accurate information. It is possible to extract oil from cores, but coring is a very expensive operation, the extraction is extremely time-consuming, and it is unlikely for normal commercial operations to give an answer to a question like this. It may be possible with the new techniques of spectroscopic examination on very small samples of crude to discover differences, but there is no doubt that oils of different composition could have been mixed in the reservoirs, and there is a body of opinion which holds that the Burgan reservoir, at least, was first filled by a migration of crude, which escaped either through faults

* Falcon, N. L. "Position of Oilfields of South West Iraq with Respect to Relevant Sedimentary Basins," "Habitat of Oil." Tulsa: Amer. Ass. Petrol. Geol.; 1958.

or through a very thin caprock. The caprock on top of Burgan is only about 100 feet thick, and if oil was in the reservoir at the time the caprock was eroded, it is quite probable that quite a lot of it did escape. It has been held that the heavy oil patches are relics of such a "lost" migration, but if that was true the heavy oil should be concentrated in the reservoir at the points of escape. The main body of the heavy crude is found at the bottom of the crude oil column, next to the oil/water contact and, if it is the relic of an earlier migration, the bulk of which escaped through the top of the reservoir, it must have been pushed from top to bottom of the oil column. It is practically immobile now, and it is difficult to imagine that it has been pushed down some hundreds of feet by new oil. The dispersed patches of heavy oil appear to be evenly distributed throughout the Wafra, Mauddud, and Burgan formations.

The effect of the seepages is again almost impossible to deduce. There is no doubt that oil would escape from the top of the reservoir rather than the bottom, and if oil was distributed as it is now, with light oil on the top, the seepage must have had the effect of losing a lot of the lighter ends. There is one consideration, which may be relevant, that on the Ahmadi structure there is no seepage. On the Magwa structure, there is a small gas seepage, with no evidence of much oil coming to the surface, although there is gas and some oil in the Eocene beds. On Burgan there is a considerable seepage, with large bitumen deposits at the surface, and it is possible that the differing densities of the crude oils left in the three reservoirs may be due to the escape to the surface of different quantities and qualities of gas and crude from them.

The only fact I can think of that could argue against that is that in the common part of the reservoir the level of heavy oil is more or less the same throughout. Had the seepage at Burgan taken off a higher proportion of the light crude, one would expect the heavy oil to have risen farther in the Burgan reservoir to replace it, and that does not appear to have happened.

T. M. W. Strong: I have had the opportunity of discussing some of these points on several occasions with Mr Fox, and also have had occasion to look into some of the very interesting problems which he has outlined this evening.

Regarding the symmetric of radial arrangement of oil quality mentioned by Mr Fox, detailed examination shows that in the NE. part of Burgan one has to go about 300 feet deeper to get the same gravity of oil as in the SW. This suggests that the oil has actually migrated from the NE., from the main geosyncline; and as it has come across Burgan, it has apparently lost light ends so that, to the SW. of the crest, at which the main leakages occurred during the uplift, a heavier crude is left behind. Going farther north the crudes are progressively lighter, the term "light" implying merely a higher proportion of light distillates.

The terms light and heavy are widely used, but may imply two different things, so that one can have two crudes with the same gravity, one having more light ends, but with heavier individual fractions, while the other may have a lower proportion of light ends while its individual fractions are lighter. Most of the Middle East commercial oils, however, are of very similar composition, fraction for fraction, and gravity variations in a given distillate cut, though present, are small; this can be seen by examining the Hempel analyses of Middle East oils as published by the U.S. Bureau of Mines. Therefore, for these oils, the terms "light" and "heavy" generally infer merely a difference in the proportion of distillates.

A plot of the distribution of oil gravities in Kuwait therefore reflects the geological history of the field with periods of original feed, loss by evaporation during uplift, and subsequent reburial and re-charging by continued feed of fresh oils from the NE.

This picture provides a possible explanation of the basal heavy oils and tar mat in Kuwait. Thus, before the cover in Kuwait was ever reduced to 100 feet, the previous oilfield would have completely burst its top and produced fractures, manjak veins, and all the seepage phenomena commonly observed in Middle East seepage areas, and this could have continued for a considerable period after reburial, with considerable thickening up of the oil before the new cover was effective. And if that is the case, one would expect to have lost at least half of the oil from the oilfield during Cenomanian time, between 60 and 70 million years ago. That condition must have lasted quite a long time, and if the oil was lost, presumably the water level must have risen, until new deposition took place and a new cover formed strong enough to hold newly increasing gas pressures arising from the entry of new oils. These had to push down all the old heavy oil and water because there was nowhere else for it to go. Therefore the tar and associated heavy oils may be regarded as representing the older degraded oil pushed down by the new oil, which entered later.

Considering further the question of oil gravities, it may be stated as a general rule that oils deficient in distillates tend to exhibit higher gravities in corresponding cuts. For instance, the narrow fraction boiling at, say, 409° C varies from 8·85 in the lightest and most paraffinic crudes to 1·00 sp. gr. or more in some of the heavier residual oils.

The gradual transference of resins to the heavier ends during migration appears to play an extremely important part, though alterations in the proportion of paraffins, naphthenes, and aromatics are important in the lighter distillate range.

It is a curious fact that many of the lightest world crudes, fraction for fraction, have migrated through long distances through very fine sediments, and the deduction is that when pores and cracks are of molecular dimensions, the condition causes a separation of molecular groups according to their shape, size, and weight, with preferential movement of the paraffins and a gradual differentiation or "fractionation" of the crude by simple physical means. The initial composition of oil (apart from its original sulphur content), as formed from the original mixed organic matter, is probably fairly constant, and always has been, so that its present composition reflects what has happened to it since. The initial sulphur content is normally high in oils from calcareous source rocks containing sulphates. These considerations can be applied to some extent to the detailed study of oil quality variation in the coarse and fine sediments in Kuwait.

Mr Fox has mentioned in the text that tars are present below the present water, and there is, of course, no proof that the water in an oilfield over a long period of time will remain static or that its present oil–water level is the lowest in the history of this or any other field.

On the tectonics side, the question of whether the Zagros or other strains and stresses could be transmitted 200 miles, this I think would be determined by the thickness of both the sediments and the basement, because even a modest 50,000 feet, or say 10 miles, of competent beds could transmit quite a stress over 200 miles.

The actual displacements in Kuwait are really very

small, because with dips of between 2° and 3° the actual compression is only one part of a thousand, *i.e.* one foot lateral compression in 1000 feet will give a 2½° fold. The vertical rise is about 40 times this, so that large vertical displacements can be caused by very small lateral compressions.

A. F. Fox: I find it difficult to imagine a large escape of oil from the Kuwait reservoirs in the Cretaceous. If the migration of crude into the reservoir started immediately after the caprock was formed and has continued at a steady rate since then, only a very small quantity of crude would have been in the reservoir at the time of the caprock erosion. It is, however, possible that migration was rapid and, as source rocks were present from the Triassic onwards, the Burgan reservoir could have been filled with oil derived from older rocks as soon as there was a competent caprock. Such oil would have had to migrate vertically, and vertical migration in an area of relatively undisturbed sediments seems considerably less likely than horizontal migration. I think it is more probable that oil was generated in rocks of the same age as those in which it is now found, rather than oil generated in the Trias migrating up through a number of horizontal barriers to find its eventual home in the Cretaceous or even in the Tertiary.

Generally speaking, there is a worldwide agreement that older oils are lighter than young oils, and one would have expected that, had there been any upward migration, with crude formed in, say, the Trias and now found in the Cretaceous, its gravity would indicate that it was Triassic oil.

If the average gravities of Middle East crudes are plotted against the ages of their reservoir beds, there is a general agreement with the worldwide relationship, the older reservoir beds containing the lighter oils. More particularly, the SE. Kuwait reservoirs do not show any abnormal departure from the general trend. I therefore believe that migration has been horizontal and must have started after the beds had been deposited. There is a considerable doubt whether in fact the forces available are sufficient to cause any migration of oil. But if they are sufficient, then movement of crude into a reservoir must be very slow and, although perhaps it is not continuing at present, it must have continued for a very long time, so that before there was an appreciable quantity of oil in the reservoir it was sealed by quite a considerable thickness of beds.

D. H. Peel: It is with some temerity that, as a mere chemist, I rise to accept the challenge put out by Dr Henson. But for the last ten years it has been my privilege to be closely associated with Mr Fox and the Kuwait Oil Company, and I have had access to the analyses of a very large number of samples withdrawn from the various Kuwait fields and from many other fields in the Middle East. So, in a sense, I am in a position to comment, if I may, on the chemical aspects which have been raised.

I agree entirely with Mr Fox's description of the Kuwait fields. I might add that the variations in crude oil quality with depth in the reservoir extend to all sorts of other features of the crude, in addition to API gravity. For instance, as one goes deeper through the structure the sulphur content of the crude rises considerably from less than 2 per cent at the top of the field to 3·6 per cent or more down at the base; the yields of distillates *v.* residues decrease as one goes deeper and there is more and more heavy residue and the quality of the residue cut at a fixed TBP temperature also changes markedly; the content of asphaltenes increases sharply, as also do the carbon residue content and the viscosity, and there is a marked increase in the vanadium content. The rates of change of quality become much steeper at the base of the structure, where the oil near the water contact often approaches a heavy tar. These changes of crude oil quality with depth are completely progressive and apply also to distillate fractions on a TBP column. The properties of these narrow boiling fractions themselves change progressively; they get heavier in specific gravity, contain more sulphur, and are more aromatic.

All of these features fit very nicely into the theory which Mr Fox has put forward which postulates the setting up of quality gradients by migration of the crude through an adsorptive medium.

Extending this picture beyond Kuwait, one finds that the crude in the fields of S. Iraq is of a type which is very similar to that of the fields in Kuwait. But the S. Iraq crudes are lighter and, broadly speaking, one finds there is a general trend for the overall average quality of the crude in the reservoir itself to become progressively heavier as one moves in a N. to S. direction through a series of reservoirs from S. Iraq (Zubair, Rumaila), through N. Kuwait (Raudhatain), to Magwa, Ahmadi, and finally to the Burgan field itself. Also this N. to S. trend in quality can even be detected by quality differences across the Burgan field itself.

My view is that the crudes of the main producing zones in S. Iraq and Kuwait are of essentially the same basic type, and hence probably of the same origin, but that the quality differences now encountered as between one field and another and also as between different depths in the same field can all be accounted for by postulating a N. to S. migration through an adsorptive medium. The crude in the Burgan sand in the Neutral Zone, is, however, an exception in being a different type of crude with quite different hydrocarbons.

In reply to Dr Henson's point about mixtures of crude, I agree that any theory of the migration and origin of oil must always be thrown to the winds if there are possibilities of mixing oils from two sources, as could happen if a new oil migrated into a reservoir still containing remnants of an entirely different crude. I can only recall one case that would appear to be a mixture of two essentially different crudes, and this is the Ratawi limestone in the Neutral Zone; this crude appears to me to be a mixture of a normal light crude and an extremely heavy asphaltic crude, and there is a marked discontinuity in many of the properties.

Finally, I would like to amplify these views on the migration and origin of oil in the Middle East by stating that to my knowledge most Middle East crude oils which are now present in *sands* show this peculiarity of a progressive change in quality with depth within the one reservoir, *i.e.* a fall in API gravity, an increase in sulphur content, a rise in viscosity, and all the other properties I have mentioned as one goes deeper into the sandstone. On the other hand, most of the Middle East crudes of which I have knowledge which are now present in *limestones* do not show any variations in quality with depth, and the crude is completely uniform throughout the reservoir. Gach Saran has already been mentioned and Agha Jari, Kirkuk, and many others could be added to this class. I would put forward the view that those oils which are now present in limestone were originally laid down in this formation and have not migrated far, whereas those now present in sandstone and showing these quality variations have probably migrated from another source.

An interesting extension of these views stems from a

consideration of the sulphur compounds present in crude oils. I believe myself that the sulphur now in the crude was not part of the original " animal," as it were, but has entered into the crude oil later on from inorganic sources. The sulphur content of the crude oil cannot be predicted from any characteristics of the source rocks, nor is it allied with any of the methods of classifying crudes in terms of hydrocarbon types, but it appears to be completely haphazard.

It is, as far as I know, a fact that Middle East crude oils which are now present in sands and which show this peculiarity of quality variation with depth within the reservoir are either completely free from hydrogen sulphide, or practically free from it, and also contain only very small amounts of mercaptans in the light distillates. On the other hand, Middle East crude oils which are now present in limestones generally contain quite high proportions of dissolved hydrogen sulphide and low boiling mercaptans. My suggestion here is that one of the main sources of sulphur in crude is from sulphate rocks which, under bacterial action, produce hydrogen sulphide, which in turn over geological time has a sulphiding action on hydrocarbons producing first mercaptans, and then heavier and more stable sulphur compounds. This process is possibly still going on in the limestone fields, but it has now ceased in the sandstone fields because the oil, which was perhaps originally formed in a limestone where there was sulphate, has since migrated into a sandstone where there is no sulphate, no bacteria, and no hydrogen sulphide.

W. S. Ault: That was an extremely interesting contribution and, as another mere chemist, I appreciated it very much. Mr Peel has anticipated a point which I was going to make, which is also a chemical one, and that is whether this chromatographic separation in sands of certain types, this preferential adsorption which is, it seems to me, quite akin to the chromatographic separation commonly used in analysis today, commonly occurs in oil sands.

The other point which intrigues me is that it is apparently held to be possible for segregation by molecular size to occur in a very long column of oil under the action of gravity over millions of years. Some years ago I heard of what was believed to be separation of large molecules in ultracentrifuges, but they were very large molecules, much bigger than anything encountered in crude oil. I wonder whether any specialist physicists have been consulted on this, in particular the people working for the Atomic Energy Authority, who have a great deal of experience of trying to separate isotopes by all sorts of methods. Do they feel that a gravitational field operating for millions of years would give molecular separation? I would be doubtful myself, because of the normal molecular movement at the fairly high temperatures underground.

A. F. Fox: I am very grateful to Mr Peel for the support he has given to my ideas.

The Wafra crude, if it is different from the crude in S. Kuwait structures, may be a separate small migration from the centre of the basin, or a locally formed body of oil. Wafra is just about on the top of a structural trend, it is the last structure in the Burgan sands before the crest of the rise at El Jauf, and it is quite possible that it is an older structure than the others, and would have trapped earlier oil.

The differences in crude oil of the same age seems to me to be much more easily achieved by factors which have affected them during their migration rather than by anything else.

With regard to the chairman's question, the idea of the separation of columns of oil is, I think, almost entirely founded on theoretical physical work, and I am not aware that anyone has succeeded in the laboratory in making such a separation; certainly, as far as I know, centrifuging does not separate anything of molecular size. The Atomic Energy Authority has not so far been consulted.

H. C. Lack: As is well known, the Burgan field in Kuwait has the advantage of operating under a very strong water-drive, and we take full advantage of it in our producing operations. I would like to ask Mr Fox if he has any idea from where this water comes. We are producing oil at a high rate nowadays, and it is being replaced by water at an almost equally high rate. Pressure considerations indicate that the original pressure in the underground reservoir was higher than would be deduced from hydraulic considerations of pressure and depth, so that the source of the water may be at a higher elevation, somewhere in the hinterland. I wonder if Mr Fox can suggest where that may possibly be.

A. F. Fox: Little is known of the aquifer or the water. It is known that the salt content of the water in the Burgan sands is high, while the connate water shows an increase in salt content from top to bottom; the pressure is also higher, but not much higher, than the normal hydrostatic head. It is possible that the caprock, which sealed in the oil at Burgan, is continuous until the aquifer is higher than the ground surface in Kuwait. The outcrop of the Burgan sands occurs somewhere in the centre of Saudi Arabia, the elevations of the outcrop areas are not known, and there is, naturally, no information on the continuity of the caprock itself. However, it is quite possible that the outcrop is sufficiently high, and the continuance of the caprock is sufficiently good, for water pressure to be due to the hydrostatic column in the Burgan sands. On the other hand, there may be a stratigraphic change to the W. of Burgan, which is sealing the water in the formation, and the pressure is a fossil pressure. If that is so, the water-drive must come from expansion of the water, although it would involve an incredibly large volume of water to give the necessary expansion; it may be that the water column is continuous the other way and the pressure is imposed from an outcrop in S. Persia. Without more information on the geology to the E. and W., which we will get only when many more holes are punched in the surface, the question is not very easy to answer.

Dr G. D. Hobson: Before commenting, there is one query I would like to raise myself, and this relates to the idea of chromatographic separation in the sand. It seems to me that the sand must be initially water-wet. But arguing that oil displaces the water, and progressively heavier hydrocarbons displace the lighter ones, then water finally displaces the oil, bothers me quite a lot, because it seems to me to be arguing both ways. I was wondering whether there was any change in physical conditions * which could account for the reversal.

* Hough, E. W., Rzasa, M. J., and Wood, B. B. (*J. Petrol. Tech.*, 1951, **3** (2), AIMME. Tech. Paper No. 3019) reported for a methane–water–stainless steel system a change from water-wet to methane-wet with rising pressure, and a reversal to water-wet on dropping the pressure. There was hysteresis. However, this system differs in several respects from that under consideration.

With regard to the other matter, the chromatographic work was done on the heavy fraction of the oils and also on extracts recovered by solvents from the shales. The most that Bonnett was able to get out of the work, which must be looked upon as exploratory, was that there was similarity in the case of some of the shale extracts, and for one of the chromatographic fractions a similarity to the corresponding fraction of one of the oils. The spectroscopic work was done only on some fractions from the extracts, not on the entire extract.* I think that there is still a little doubt as to the final interpretation of all of the similarities. The similarities may be due in part to looking at the same fraction, and how far they have solved the problem of the origin of the oil still remains to be determined. They need to be considered in conjunction with other information.

W. S. Ault: Could I add a supplementary question to that? Must the sand be wet with water first?

A. F. Fox: No, I do not think it is. It is generally assumed that because we are dealing with marine sediments, they are water-wet. But the change from water-wetness to oil-wetness in the reservoirs is receiving quite a bit of attention now, and it has only come to light very recently that sands which are thought to be oil-wet in a reservoir can change to being water-wet as a result of water displacement of the crude. I think this has come to light only because of various developments in electric logging quite recently, and not very much is known about it. But if it can happen in that way, I can see no reason why it should not happen in reverse, and that sand grains which we will say are water-wet, if the concentration of oil round them is sufficiently great, might become oil-wet.

W. S. Ault: I find that rather difficult to follow, unless you postulate that there is some surface active material in the oil, because sand is a fairly polar material and water is a more highly polar material than oil, so I would have backed the water every time.

Do you have to postulate that the sand is first of all water-wet? Could it be sand formed by erosion, carried by the wind on top of the marine sediments? Is that geologically possible?

A. F. Fox: It is possible but unlikely.

W. S. Ault: The other question is: do you have to assume water elutriating this column? Could it not be self-elutriating by further supplies of the original crude coming in below? Wide-range crude comes in at the bottom and one is first of all going to get light hydrocarbons sitting on the sand, then getting pushed off and going farther up and heavier ones displacing them, and so on. By the time the whole column is filled up, would one not end up with lighter crude at the top and heavier down below?

Dr G. D. Hobson: I am afraid I cannot answer that, but I would stress the point that in building up the original oil column, there is displacement of water which was occupying the pores. That is, in getting the hydrocarbons in, it is necessary to displace water from the pore space, irrespective of whether the water is adsorbed on the mineral or not. It is not a question of introducing hydrocarbons into air space or anything like that.

May I just add one point? There was an enquiry about the ultracentrifuge. There is a paper by Witherspoon, who has done quite a lot of work with an ultracentrifuge, and has found some evidence of gravitational separation on such occasions, as well as some separation of asphaltic material in a centrifuge.

G. W. Halse: When I was in Kuwait, several years before oil was discovered there, the Sheik of Kuwait sent me out with a guide to see an oil seepage. What we found were little blobs of bitumen on the sand, which the Arabs with me immediately started to eat. We also saw footmarks of small fox, gazelle, or some such animals which had apparently been doing the same. There was no apparent reason for the bitumen to be there and, in those days, if a guide was sent out and failed to carry out the ruler's order, he was in danger of execution.

This bitumen was, I believe, about 10 to 15 miles S. of Kuwait, and I would like to ask Mr Fox if there are any seepages in that vicinity. If not, the presumption is that the guide planted the bitumen in a convenient position in self preservation.

A. F. Fox: There are three seepages which Mr Halse might have found. There is one at Madaniyat, which is a gas seepage. He would have noticed that—it is very pungent gas. As far as I remember, there is no oil on the surface. There is quite a bit of stuff which is rather like Gatch-i-Tarush of rock and gypsum. At Magwa there is another gas seepage, with no oil on the surface. But at Burgan there is a large quantity of solid bitumen on the surface.

Prof. S. E. Hollingworth: If we may revert for a moment to the geological–stratigraphical aspects of the paper and away from reservoir physics, I would like to comment on Dr Henson's remarks that were critical of the lagoonal origin hypothesis of the anhydrites. There are many examples of marine sediments attributed to a so-called lagoonal environment involving the deposition of substances whose constituents are present in a very small proportion in sea water, or concentrations well below normal saturation level. Marine iron ores are believed to be lagoonal, and so are anhydrites, some limestones, and certain types of shale. Marine phosphates, on the other hand, are not included. For the latter, large-scale free circulation and a sloping sea floor involving specialized environment has been postulated and widely accepted.

Possibly the lagoonal hypothesis has been somewhat overstressed.

In the case of iron ores, when one considers the iron content of sea water, of the order of a few parts to a million, it is clear that a very substantial volume of water is involved, and some special conditions in a shelf area of sedimentation rather than a lagoon may well provide the answer.

Beds hundreds of feet thick composed entirely of anhydrite, produced from sea water, also require very large quantities of water to maintain the supply of calcium sulphate and avoid precipitation of more or less soluble salts in solution. Some type of shelf is again more appropriate and more compatible with the lack of evidence of adequate barriers.

D. H. Peel: A further point regarding the Neutral Zone crude from Burgan sands at Wafra. I have

* Bonnett, B. "Organic Matter in Fine-Grained Rocks, and its Relation to the Origin of Petroleum." Ph.D. thesis, University of London, 1958.

already mentioned that its quality presents an anomaly, but it could, of course, be another case of a mixture. I should also mention the case of the Safaniya field in Saudi Arabia. The crude from the Burgan sands here is heavier than the average crude from the Burgan field in Kuwait, but quality-wise it is of the same basic type and one could match the quality of Safaniya crude in every detail that we have been able to test, if one collected a sample at a deep enough level from the Burgan field in Kuwait.

H. C. Lack: I would like to ask Dr Hobson if he knows what was the length of time of the period for this supercentrifuging in these experiments.

Dr G. D. Hobson: I cannot quote the time, but I will give a reference. It is an Illinois State Geological Survey publication, and the paper is by P. A. Witherspoon.*

I think that the time was of the order of hours or days,† but I cannot be sure now.

H. C. Lack: I ask this because I think Mr Peel will remember that some years ago work was done at Nottingham University, on, I think, an artificial crude, made up of various fractions, and there it was not possible to detect any gravity segregation by centrifuging. I was wondering whether the experiments were not conducted long enough. However, this is described as supercentrifuging. I wonder how accurately centrifuging can simulate effects taking place during geologic ages.

A. F. Fox: On the centrifuging, Dr Hobson mentioned that there was some separation of solid asphalt.

Dr G. D. Hobson: Yes, asphalt-type material was observed.

A. F. Fox: That seems to suggest that there might have been some chemical action going on, and the separation might have been due to it, rather than to the pure centrifuging.

W. S. Ault: It could have been a colloidal or semi-colloidal suspension, too.

A. F. Fox: Yes. I believe that asphalt will precipitate when crude comes into contact with brine, so maybe agitation was sufficient to bring it down.

Mr Peel's point on Safaniya is very interesting, but I think that it is doubtful if it was the same migration of crude. It would indicate to me another argument for the common origin for all the crudes in the area, rather than being a crude on the same line of migration.

The meeting then closed with a unanimous vote of thanks to the author.

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* Witherspoon, P. A. "Studies on Petroleum with the Ultracentrifuge." Illinois State Geological Survey, Report of Investigations 206 (1958).

† The duration of the centrifuge runs ranged from an hour to 12 days; many runs exceeded two days.

THE CONSISTENCY OF SEMI-FLUID GREASES *

By J. O. CLIFFE † and J. W. PEARSON ‡

SUMMARY

The value of various laboratory tests for measuring the consistency of " semi-fluid " greases has been assessed by correlating the results of these tests with subjective judgments of consistency by a " consumer panel " of nine observers.

A restricted statistical analysis of the results showed that the consistency of most of the " semi-fluid " greases examined could be adequately controlled using one or other of two simple tests. For the stiffer grades, the IP penetration offered adequate control, while the softer grades were best controlled by using a simple plunger rheometer designed for measuring the apparent viscosity of non-Newtonian materials over a range of apparent rates of shear. With only one grease were these tests unsuitable—this was a grease of low yield value based on a very viscous oil blend.

The MacMichael viscometer and the tapered-hole disk penetrometer described by Hotten and Birdsall were found to be of very limited application. Other forms of cone or cylinder penetration methods were found to offer no advantages either in scope or convenience over the IP method.

A " universal cone " developed in the U.S.A. after this work was done should extend the range covered by the normal IP penetration. It is still unsuitable for very soft semi-fluid greases.

INTRODUCTION

From time to time it becomes apparent to grease suppliers that their methods for controlling the consistency of semi-fluid greases are not ideal. It appears either that the property measured by existing control methods is not that measured by the customer in his assessment of " consistency " or that the existing specification limits applied to production are insufficiently restrictive. It may be argued that a consumer's subjective assessment of consistency is largely irrelevant, and that the important feature is the relation of rheological properties to those required by the application. Nevertheless, if a consumer can subjectively detect variations in the materials supplied he may be unwilling even to use the material in his equipment.

Before describing the present work, which was designed to improve this situation, it is perhaps desirable to define the terms " consistency " and " semi-fluid " greases. " Consistency " is that quality of a grease usually assessed subjectively and commonly described as hardness, thickness, or body. In rheological terms, consistency is probably a complex function of yield value (*i.e.* the minimum stress required to produce plastic deformation), viscosity at low rates of shear, and elasticity. Where several factors are involved in a subjective measurement it is not surprising that different observers often give conflicting reports on the consistency of grease samples, and for some time attempts have been made to measure consistency in a standard manner. The method most widely accepted at present is the cone-penetration measurement standardized by the American Society for Testing Materials (ASTM) and the Institute of Petroleum (IP). This method forms the basis of the consistency classification proposed by the National Lubricating Grease Institute of America (NLGI).

The " semi-fluid " greases are the soap/oil mixtures of soft consistency which lie above the upper limits set by the NLGI penetration classification, *i.e.* softer than an ASTM worked penetration of 385. In the U.K., however, the term is usually extended to cover greases softer in worked penetration than 350, the maximum limit set by the IP for the IP penetration method IP 50/56, thus including greases defined by the NLGI as No. 0 grade. There is also a tendency to include within the definition certain greases which fall outside the scope of semi-fluid greases described above, but are nevertheless of similar composition. For example, Grease F (Table I) is too hard to fall within the above consistency limits for semi-fluid greases. It was included in the present work because it differs only in soap content from Grease E, a " true " semi-fluid grease. There are also semi-fluid grades which, though appearing stiff because they contain a highly viscous oil, have only a low resistance to deformation, *e.g.* Grease C, which contains a substantial proportion of bitumen.

The present paper describes work done on the semi-fluid greases described in Table I to establish levels of consistency, as assessed subjectively by a consumer, and to relate these levels to laboratory tests which purport to measure some aspect of consistency.

EXPERIMENTAL

(1) *Greases Examined*

Representative greases, selected from those manufactured at a particular grease plant, were chosen for the present investigation. Brief details of the greases examined are shown in Table I.

Samples of the various greases were obtained from normal plant production. The number of samples of each grade varied from twelve for the most important greases to four for Grease A, which is only manufactured infrequently. Several samples of Grease E

* MS received 12 November 1958. † Shell Oil Company of Canada Ltd. ‡ " Shell " Research Ltd.

were also prepared in the laboratory, the soap content being varied in an attempt to give a graded variation in consistency. This attempt proved unsuccessful,

TABLE I

Grease	Soap type	Approx oil viscosity Red. I at 140° F, sec.	Additives
A	Aluminium	550	Tackiness additive
B	Calcium	1,000	None
C	Calcium	20,500	None
D	Calcium	470	Graphite
E	Aluminium	1,100	None
F	Aluminium	1,100	None
G	Calcium	100	None

but the data are still of value in relating " consumer reaction " to laboratory measurements of consistency.

(2) *Consumer Consistency Ratings* (" *Subjective Hardness Ratings* ")

The consistency ratings likely to be accorded to the various samples of a given grade by an " average consumer " on the basis of subjective judgments were determined by a panel of nine judges. The judges were at liberty to form their opinions by tactual and visual methods, *e.g.* tactual assessments of hardness or viscosity and visual ratings of flow characteristics. Each judge acted independently of the others and examined the samples within a given grade by the method of paired comparisons, *i.e.* each sample of the grade was compared directly with every other sample of the same grade. Pairs of samples were examined in a randomized order and ranked according to the following scale:

	Score given to A
Sample A is much harder than Sample B	+2
Sample A is slightly harder than Sample B	+1
No difference	0
Sample B is slightly harder than Sample A	−1
Sample B is much harder than Sample A	−2

This method of assessment permitted some idea of the reliability of the judges to be obtained by noting the number of " circular triads " included in each judge's scores. These consist of sets of comparisons of the form:

A is harder than B
B ,, ,, C
C ,, ,, A

which are obviously contradictory. The criterion lost some of its usefulness because it could not take into account " no difference " judgments, but it was still to some extent a measure of reliability.

The small number of judges precluded the exclusion as unreliable, for any particular grease, of any judge who had formed such sets of contradictory judgments for the grease, so one triad was considered (arbitrarily) to be allowable. If more than one triad was found among a judge's assessment for a given grade his judgment was considered unreliable and his score was omitted when the ratings for the grade were computed.

TABLE II

Summary of Present Methods for Determining Consistency of Semi-Fluid Greases (based on Ref. 1)

Method	Type of apparatus	Advantages	Disadvantages
Lightweight cone	Used in ASTM penetrometer	Convenient, low cost	Insensitive; floats in soft greases
Potter–McLennan perforated disk	Used in ASTM penetrometer	Convenient, low cost	Range limited to the harder semi-fluid greases
Gardner mobilometer	Falling perforated disk or cone under various loads	Wide range	Inconvenient to use; hard to clean; specifications complicated
MacMichael viscometer	Rotational viscometer	Wide range	Expensive; complicated operation and specifications
Brookfield viscometer *	Rotating spindle mounted on torsion head	Wide range	Expensive; complicated specifications
Simplified pressure viscometer	Grease forced through capillary by gear pump or air pressure	Wide range, fixed shear rate	Expensive; operation complicated because of Bourdon gauge; hard to clean
Brabender viscometer	Torque measured on paddle immersed in cup of grease	Automatic recording, wide range	Expensive; complicated specifications and operation
Tapered hole disk	Used in ASTM penetrometer	Convenient, low cost	Hard to clean
Plunger rheometer	Falling plunger under various loads	Convenient, low cost	Range limited to the softer semi-fluid greases
Cylinder penetration	Falling cylinder under constant load	Convenient, low cost	Insensitive; floats in soft greases; long duration of test

* Brookfield Synchro-electric Viscometer, Model LV, Brookfield Engineering Laboratories, Sharon, Mass., U.S.A.

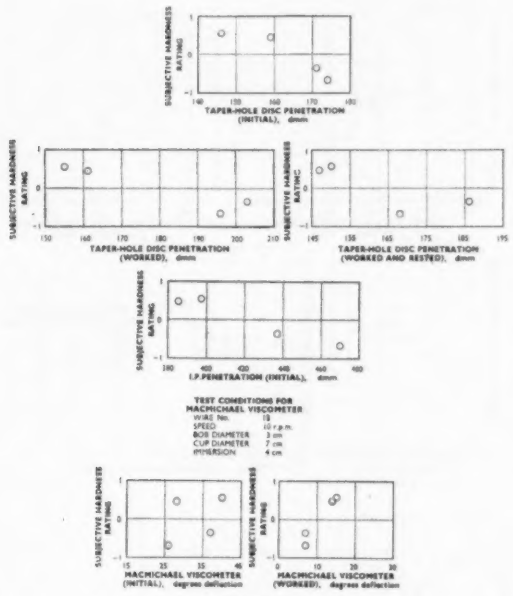
SUBJECTIVE HARDNESS RATING
TAPER-HOLE DISC PENETRATION (INITIAL), dmm
TAPER-HOLE DISC PENETRATION (WORKED), dmm
TAPER-HOLE DISC PENETRATION (WORKED AND RESTED), dmm
I.P.PENETRATION (INITIAL), dmm
TEST CONDITIONS FOR MACMICHAEL VISCOMETER
WIRE No. 18
SPEED 10 r.p.m.
BOB DIAMETER 3 cm
CUP DIAMETER 7 cm
IMMERSION 4 cm
MACMICHAEL VISCOMETER (INITIAL), degrees deflection
MACMICHAEL VISCOMETER (WORKED), degrees deflection

FIG 1

GREASE A

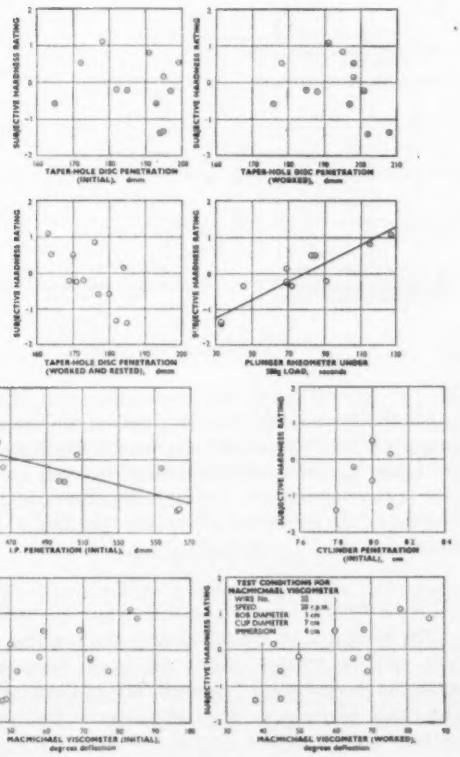
SUBJECTIVE HARDNESS RATING
TAPER-HOLE DISC PENETRATION (INITIAL), dmm
TAPER-HOLE DISC PENETRATION (WORKED), dmm
TAPER-HOLE DISC PENETRATION (WORKED AND RESTED), dmm
PLUNGER RHEOMETER UNDER 100g LOAD, seconds
I.P. PENETRATION (INITIAL), dmm
CYLINDER PENETRATION (INITIAL), cm
TEST CONDITIONS FOR MACMICHAEL VISCOMETER
WIRE No. 22
SPEED 30 r.p.m.
BOB DIAMETER 3 cm
CUP DIAMETER 7 cm
IMMERSION 4 cm
MACMICHAEL VISCOMETER (INITIAL), degrees deflection
MACMICHAEL VISCOMETER (WORKED), degrees deflection

FIG 2

GREASE B

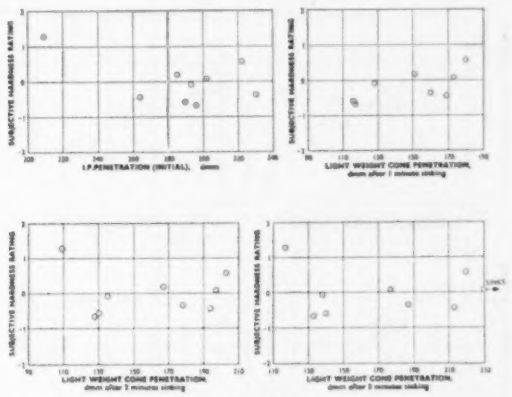

FIG 3

GREASE C

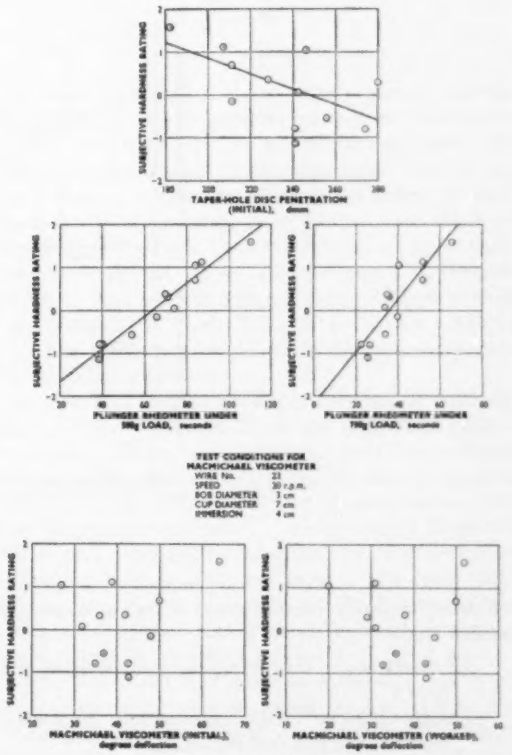
SUBJECTIVE HARDNESS RATING
TAPER-HOLE DISC PENETRATION (INITIAL), dmm
PLUNGER RHEOMETER UNDER 100g LOAD, seconds
PLUNGER RHEOMETER UNDER 700g LOAD, seconds
TEST CONDITIONS FOR MACMICHAEL VISCOMETER
WIRE No. 22
SPEED 30 r.p.m.
BOB DIAMETER 3 cm
CUP DIAMETER 7 cm
IMMERSION 4 cm
MACMICHAEL VISCOMETER (INITIAL), degrees deflection
MACMICHAEL VISCOMETER (WORKED), degrees deflection

FIG 4

GREASE D

(3) *Laboratory Measurements of Consistency*

Several methods have been described for measuring the consistency of semi-fluid greases, and a useful summary has been published.[1] This summary is given in Table II, together with additional methods which have also been used. From the list, the

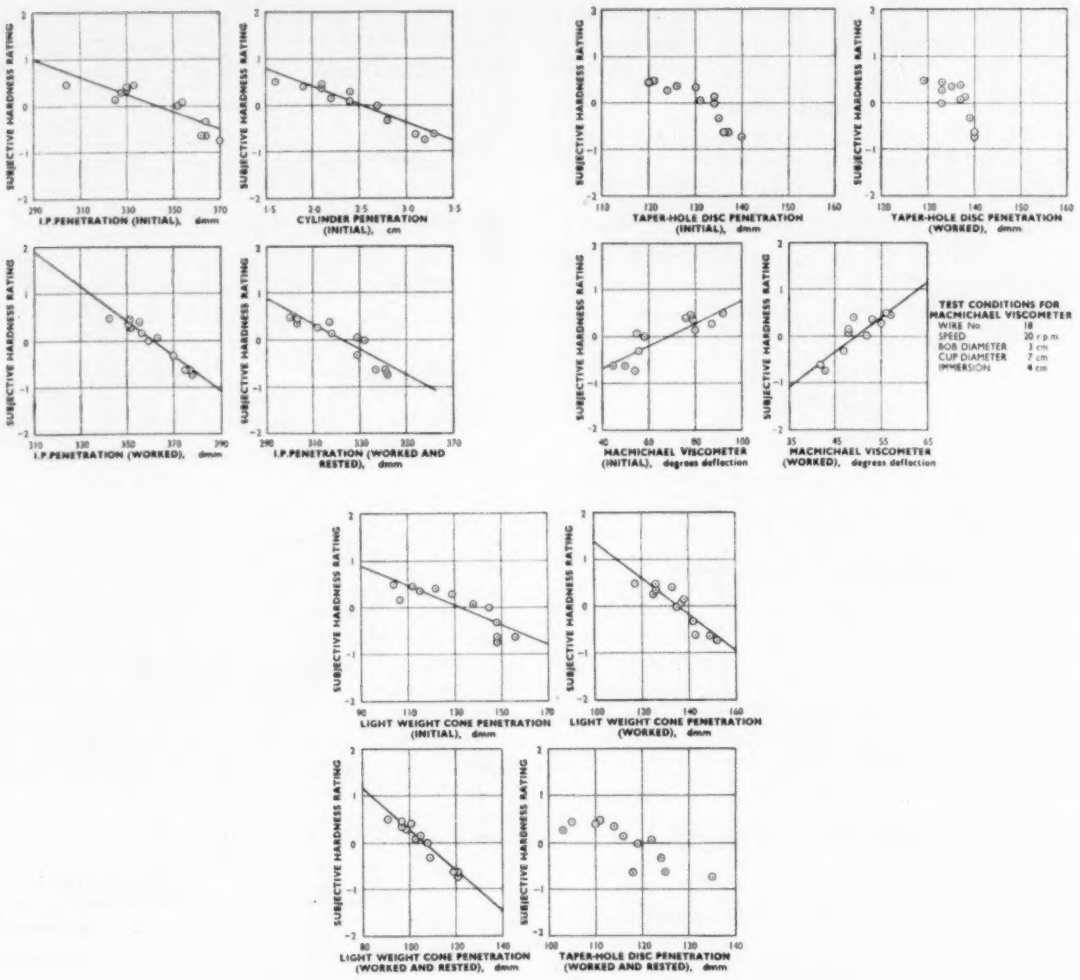
SUBJECTIVE HARDNESS RATING
I.P.PENETRATION (INITIAL), dmm
CYLINDER PENETRATION (INITIAL), cm
TAPER-HOLE DISC PENETRATION (INITIAL), dmm
TAPER-HOLE DISC PENETRATION (WORKED), dmm
I.P.PENETRATION (WORKED), dmm
I.P.PENETRATION (WORKED AND RESTED), dmm
MACMICHAEL VISCOMETER (INITIAL), degrees deflection
MACMICHAEL VISCOMETER (WORKED), degrees deflection
TEST CONDITIONS FOR MACMICHAEL VISCOMETER
WIRE No. 18
SPEED 20 r.p.m.
BOB DIAMETER 3 cm
CUP DIAMETER 7 cm
IMMERSION 4 cm
LIGHT WEIGHT CONE PENETRATION (INITIAL), dmm
LIGHT WEIGHT CONE PENETRATION (WORKED), dmm
LIGHT WEIGHT CONE PENETRATION (WORKED AND RESTED), dmm
TAPER-HOLE DISC PENETRATION (WORKED AND RESTED), dmm

FIG 5

GREASE E

following methods were selected as being worthy of further study:

IP penetration, unworked, worked, worked-and-rested.
Lightweight 90° cone penetration, unworked, worked, worked-and-rested.
Cylinder penetration.
Tapered hole disk penetration.[1]
Viscosity by MacMichael viscometer.
Viscosity by plunger rheometer.

Consideration was given in selecting the methods to their suitability for use in a plant control laboratory and the availability or simplicity of the apparatus. Where adequate references are not available, details of the methods used are given in the Appendix.

All the tests could not be used on all the greases examined because of the physical limitations of the methods, *e.g.* with all batches of Greases D and G and some batches of Grease B the IP cone hit the bottom of the container within the specified 5 seconds free fall.

(4) *Test Results*

The results of all the laboratory tests carried out on the various greases examined have been plotted against the subjective hardness ratings determined by the panel of judges. For this purpose the hardness ratings are expressed as the average of the marks awarded to each sample, and it follows that the more positive the rating, the harder the grease. The

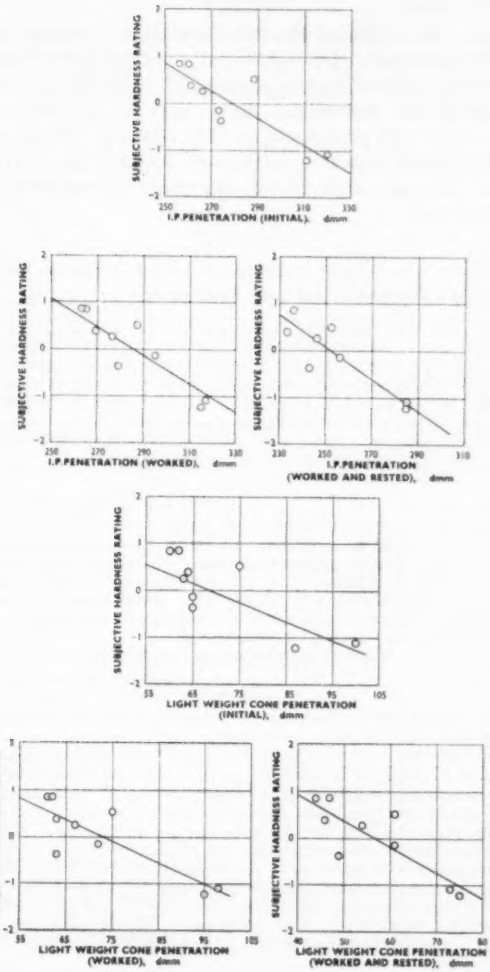
SUBJECTIVE HARDNESS RATING
I.P. PENETRATION (INITIAL), dmm
I.P. PENETRATION (WORKED), dmm
I.P. PENETRATION (WORKED AND RESTED), dmm
LIGHT WEIGHT CONE PENETRATION (INITIAL), dmm
LIGHT WEIGHT CONE PENETRATION (WORKED), dmm
LIGHT WEIGHT CONE PENETRATION (WORKED AND RESTED), dmm

FIG 6

GREASE F

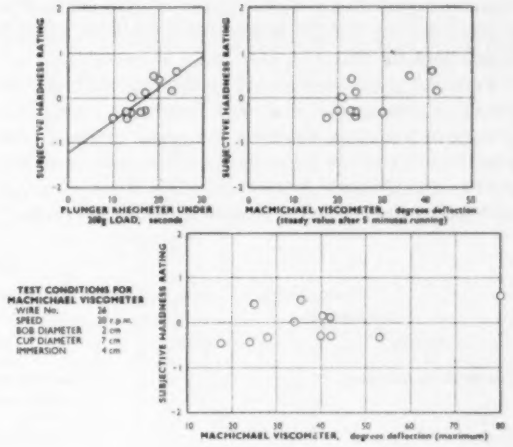
SUBJECTIVE HARDNESS RATING
PLUNGER RHEOMETER UNDER 200g LOAD, seconds
MACHICHAEL VISCOMETER, degrees deflection (steady value after 5 minutes running)
TEST CONDITIONS FOR MACHICHAEL VISCOMETER
WIRE No. 26
SPEED 20 r.p.m.
BOB DIAMETER 2 cm
CUP DIAMETER 7 cm
IMMERSION 4 cm
MACHICHAEL VISCOMETER, degrees deflection (maximum)

FIG 7

GREASE G

results of this comparison are shown in Figs 1 to 7. In these figures, actual lines have been drawn only in those cases where an obvious correlation exists between the subjective hardness rating and the laboratory test results.

DISCUSSION

(1) *The Relationship between Subjective Hardness Ratings and Laboratory Tests*

The small number of samples examined precluded any rigorous statistical investigation of the results, and the examination was restricted to simple analyses of regression. On this basis Table III has been compiled, in which the relative values of the various test methods as predictors of grease consistency are indicated.

It must be stated that the data reported here are insufficient to make positive predictions as to the " best " laboratory test for any particular grade. To keep the programme within reasonable bounds, no more than a maximum of twelve batches of each grade could be included, these having been taken at random in the hope that the widest range of consistency expected to be met in plant production might be covered. Before any finer distinction than that outlined in Table III can be drawn, reliable estimates of the reproducibility of the laboratory tests will have to be obtained.

It is apparent that no single test can be used for the consistency control of all the semi-fluid greases examined. There are, however, two tests which between them give some measure of control for most of the greases. These are the IP penetration test and the plunger rheometer. In the case of Grease C, the control exercised by means of the IP penetration is likely to be only of limited value. The lightweight cone penetration is generally as satisfactory as the IP penetration, but the IP method, which is already standardized, is obviously preferable.

After this work had been completed, a universal penetration cone was proposed in the U.S.A. for controlling the consistency of all greases.[2] This is based on the ASTM/IP cone, but the rate of penetration beyond about 450 dmm is reduced by narrowing the annular clearance between the cone and the container. A brief examination suggests that this cone would extend the usefulness of the IP type penetration to include Greases B and perhaps D. The new cone still hits the bottom of the container within the specified 5 seconds fall when testing Grease G or certain very soft aircraft greases developed since this work was started. Although these greases

represent a relatively small offtake, they must still be controlled: the plunger rheometer is the most suitable instrument for this.

Penetration measurements by the tapered hole disk correlate reasonably well with hardness ratings for greases in the range covering the upper limits of cone penetration and the lower limits of viscosity measurements, *e.g.* Greases B and D, while there is rough correlation in the case of Grease A, which also falls in this consistency range. Since Greases B and D can be as well controlled by the plunger rheometer, while Grease A can be as well controlled by IP penetration, there is no justification for introducing the additional instrument in these cases.

It is noteworthy that the field of application of the MacMichael viscometer to semi-fluid greases is rather restricted. Of the greases examined in the present series its use could only be justified for Grease A.

The cylinder penetration method appears a suitable control for Grease E, but this is a 20-hour test and the IP penetration is therefore preferred.

Conclusions

1. The stiffer grades of "semi-fluid" greases can be realistically and conveniently controlled by penetration measurements according to IP 50/56, even though the penetration figures may exceed the maximum of 350 prescribed by this method. There are indications that the method can be used even up to the absolute limit of the apparatus (namely, about 600 dmm).

TABLE III

Classification of Laboratory Methods as Predictors of the Consistencies of Semi-Fluid Greases

Grade	Possible	Doubtful	Unlikely	Unsuitable
Grease A (4 batches)		MacMichael viscometer † Tapered hole disk † penetration IP penetration †		Plunger rheometer Lightweight cone penetration Cylinder penetration
Grease B (12 batches)	Plunger rheometer IP penetration *	Tapered hole disk penetration MacMichael viscometer		Lightweight cone penetration Cylinder penetration
Grease C (9 batches)			IP penetration Lightweight cone penetration	Cylinder penetration MacMichael viscometer Tapered hole disk penetration Plunger rheometer
Grease D (12 batches)	Plunger rheometer Tapered hole disk penetration		MacMichael viscometer	IP penetration Lightweight cone penetration Cylinder penetration
Grease E (12 batches)	IP penetration Lightweight cone penetration MacMichael viscometer Cylinder penetration	Tapered hole disk penetration		Plunger rheometer
Grease F (9 batches)	IP penetration Lightweight cone penetration			Cylinder penetration Plunger rheometer Tapered hole disk penetration MacMichael viscometer
Grease G (12 batches)	Plunger rheometer	MacMichael viscometer		IP penetration Lightweight cone penetration Cylinder penetration Tapered hole disk penetration

* Four batches were too soft for determination.
† These methods cannot be rated more highly because of the very limited data.

2. For the softer greases the use of the plunger rheometer provides a realistic control of consistency.

3. No firm recommendations can be made for controlling Greases A and C, although IP penetration should afford some control.

4. The MacMichael viscometer, which is frequently used for some semi-fluid greases, is of little value for controlling consistency.

5. Penetrations of the tapered hole disk or the cylinder are realistic methods of controlling consistency for certain greases, but are of only limited application.

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[1] Hotten, B. W., and Birdsall, D. H. *Analyt. Chem.*, 1952, **24**, 892. (Since the work described in the present report has been completed, this method has been published "for information only" in the ASTM "Standards on Petroleum Products and Lubricants," Nov. 1955, Appendix VIII.)

[2] U.S. Patent 2,794,339, "Penetrometer Cone for All Greases." L. C. Brunstrum and A. C. Borg, assignors to Standard Oil Co., Chicago.

APPENDIX

Test Methods used for the Control of "Semi-Fluid" Greases

(1) *IP Penetration*

The apparatus and procedure used were identical with that described in the IP 50 method for penetration. "Initial" penetrations were determined by transferring the sample to the worker pot with the minimum of working and then determining the penetration. "Worked" penetrations were determined on the same sample after 60 strokes working and "rested" penetrations were determined on the worked sample after 24 hours resting at the test temperature (25° C).

(2) *Lightweight Cone Penetration*

A 90° duralumin cone and special lightweight plunger assembly (total moving weight 15 g) were used in a penetrometer with a slightly modified clamping device. The cone was lowered into the sample at a standard rate of 0·5 mm/sec until it floated freely. The depth of penetration (in dmm) was noted after 60 seconds free floating. The terms "initial," "worked," and "rested" penetrations had the same significance as in the IP penetration tests.

(3) *Tapered Hole Disk Penetration* [1]

The apparatus consisted of a duralumin disk bored with twelve tapered holes and mounted on a pointed steel spindle passing through the centre of the disk. The point of the spindle projected through the disk by a distance of 1 cm (100 dmm). When mounted in a standard penetrometer shaft, the total moving weight (disk and shaft) was 150 g.

For measuring grease consistency, the disk was used in a manner analogous to the IP penetration test, a penetration time of 5 seconds being allowed. The design of the disk was such that only penetrations appreciably greater than 100 dmm were of any significance, this value representing the length of the projecting shaft. At a penetration of 100 dmm the disk itself was merely resting on the surface of the grease.

(4) *Plunger Rheometer*

This apparatus, which is shown diagrammatically in Fig 8, consisted of an upright brass cylinder containing a centrally located plunger which could be loaded by means of added weights up to a maximum weight of about 750 g. The sample was filled into the cylinder and the plunger was allowed to sink through it under a known load through a fixed distance of 10 cm. For routine control determinations, the time of fall (seconds) under a specified total moving weight was reported.

By calibration of the instrument against oils of known viscosity it was possible to calculate the apparent viscosity of the material for any given load and rate of fall. From the geometry of the instrument it was also possible to calculate the corresponding apparent rate of shear. Thus, by making several determinations under various loads the apparent

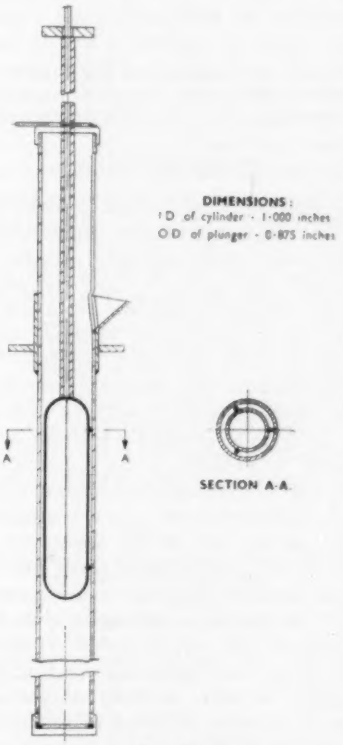

FIG 8

PLUNGER RHEOMETER AS USED IN THE PRESENT INVESTIGATION (not to scale)

viscosity of the sample at a standard rate of shear could be calculated by interpolation or extrapolation of the curve of logarithm of apparent viscosity against logarithm of apparent rate of shear.

(5) *Cylinder Penetration*

The sample was contained in a tin of specified size and a hollow aluminium cylinder with a flat solid bottom (weight 10·52 g) was allowed to sink vertically into the grease. After 20 hours settling, the depth of immersion (cm) was read off from graduations on the outside of the cylinder.

ALUMINA AS A COLUMN PACKING IN GAS CHROMATOGRAPHY *

By C. G. SCOTT †

SUMMARY

A study of the adsorptive properties of alumina in terms of gas-chromatographic data has shown that this adsorbent can be used as a column packing for the separation of hydrogen–hydrocarbon gas mixtures at room temperature. The relationship between the data and surface properties of the alumina is also discussed.

INTRODUCTION

Gas chromatography has become an established technique for the analysis of refinery gas streams. Of the two types of such chromatography, namely, gas–liquid and gas–solid, the former is most favoured because symmetrical elution peaks are obtained and the separation can be carried out at room temperature. Unfortunately, with partition columns, hydrogen cannot be separated from methane, and with the inclusion in most refineries nowadays of a reforming unit, the analysis of streams containing hydrogen is of increased importance. One procedure for such mixtures is to supplement the analysis of the hydrocarbons on a partition column by a hydrogen/methane separation carried out on an adsorption column.[1] Alternatively, it is possible to carry out the entire analysis on an adsorption column using some form of stepwise or programmed heating to elute the higher boiling hydrocarbons which would otherwise be retained on the column for too great a length of time.[2]

This paper describes a study of the adsorptive properties of alumina in terms of retention volume data for ethane, ethylene, and propane from which it has been possible to deduce the necessary column packing characteristics to obtain a complete separation of hydrogen and 14 hydrocarbons ranging from methane to *n*-pentane on one comparatively short column operating at room temperature. The relationship of the gas chromatographic data to the surface properties of the alumina is also discussed.

EXPERIMENTAL

The work was carried out at room temperature using a hydrogen flame detector with nitrogen as carrier gas. Preliminary experimental work showed that for a 1-ml gas sample (three components) injected on to a 4 mm i.d. or 6 mm i.d. column, the retention volumes reported in ml of carrier gas (measured from a hydrogen peak and corrected for pressure drop across the column) per gram of adsorbent could be used to characterize the activity level, at a given temperature, of a weak adsorbent from which near symmetrical elution peaks are obtained. These values are referred to hereafter as U_g° values.

The alumina used was British Drug Houses Ltd "Chromatographic Grade," intended for use in liquid elution chromatography. It has a Brockmann grading of I–II corresponding to a water content of nearly 3 per cent by weight. This material was sieved to give a 100–150 B.S. mesh fraction, which, when used as a column packing, gave U_g° values of 1·85, 3·90, and 8·50 for ethane, ethylene, and propane respectively. Fig 1 shows the data in the more familiar form of a chromatogram.

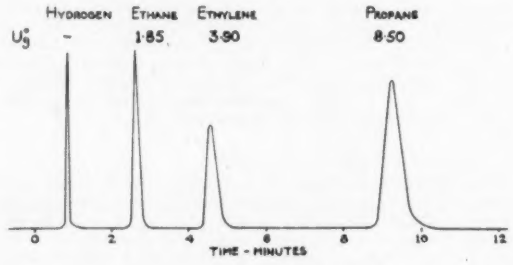

Fig 1

SEPARATION USING ALUMINA AT INITIAL ACTIVITY LEVEL
Column: 6 ft × 4 mm i.d., 30 g alumina 100–150 mesh 15° C
Carrier gas: Nitrogen, 30 ml minute (corrected)

The activity of the starting material was increased by subjecting separate portions for a one hour period to temperatures ranging from 200° to 1000° C. The activity of another portion of the starting material was reduced in stages by allowing it to sorb moisture. The U_g° values for ethane, ethylene, and propane were determined at each of the activity levels.

The results obtained on the aluminas heated at temperatures up to 500° C, together with the results on the material whose activity was lowered, are shown in Fig 2. Fig 3 shows the values obtained for the aluminas heated at temperatures ranging from 200° to 1000° C.

RESULTS AND DISCUSSION (Fig 2)

In Fig 2, the logarithms of the U_g° values for ethane are joined to the corresponding values for propane. The corresponding logarithm of the U_g° values for ethylene are also indicated.

* MS received 3 December 1958.

† Lobitos Oilfields Limited.

The plot Activity " A " shows the values obtained on the starting material. The value for either of the saturates ethane or propane indicates the activity level of this alumina and the position of the unsaturated ethylene relative to the ethane and propane axes indicates the polarity of the alumina.

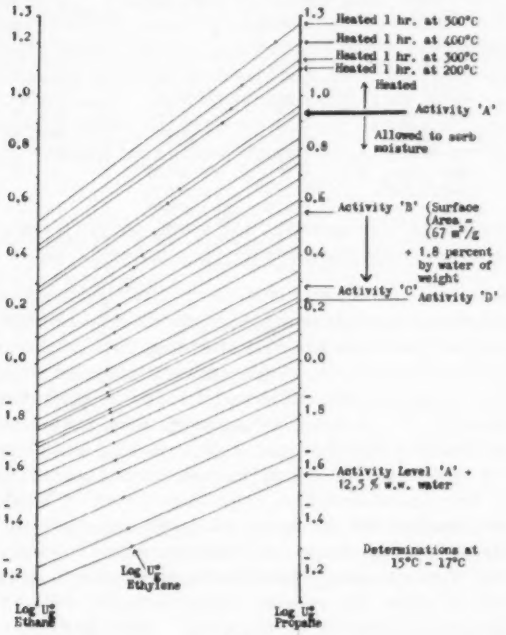
Heated 1 hr. at 500°C
Heated 1 hr. at 400°C
Heated 1 hr. at 300°C
Heated 1 hr. at 200°C
Heated
Activity 'A'
Allowed to sorb moisture
Activity 'B' (Surface Area = (67 m²/g + 1.8 percent by water of weight
Activity 'C'
Activity 'D'
Activity Level 'A' + 12.5 % w.w. water
Determinations at 15°C - 17°C
Log Ug° Ethylene
Log Ug° Ethane
Log Ug° Propane

FIG 2

CHANGE IN POLARITY WITH ACTIVITY LEVEL OF ALUMINA

As the water content of the alumina is reduced in stages by heat treatments at 200°, 300°, 400°, and 500° C the polarity of the alumina increases at a much greater rate than the activity and indicates that, as water is driven off, highly active polar sites are made available for hydrocarbon adsorption.

When water is sorbed by the starting material, the first uptakes cause the polarity to decrease at a greater rate than the activity until a minimum polarity is reached (Activity " C "). Further uptakes of water, although still causing the activity to decrease, tend to increase the polarity slightly. It was noted, by comparing the results obtained for this particular alumina with the results for another less dense alumina, that the amount of water required to reduce the alumina to a state of minimum polarity was a function of surface area.

Data supplied by the manufacturer on the alumina used for the work showed that the additional water required to bring the alumina to a state of minimum polarity approximated to the amount generally required by this material to cover the surface with a monolayer of water.

Cornelius *et al* [3] have shown that the weight of water sorbed by a dehydrated or nearly dehydrated alumina cannot be used to determine its surface area because the initial uptake of water which is accompanied by extremely high heats of sorption is probably chemisorbed water. They found that the weight of water required to give a monolayer coverage by B.E.T. procedure was some 30 per cent greater than the figure calculated from nitrogen monolayer coverage by B.E.T. method. Correlation of surface area of the alumina with gas chromatographic data was therefore carried out on a sample of alumina whose surface was already partly covered with a monolayer of water.

The $U_g°$ values for the alumina used for the determination were as Activity " B." The surface area which was still available for water sorption was determined by B.E.T. procedure using nitrogen, the preliminary evacuation being made at room temperature and note being made of water lost during evacuation. Although the results obtained by this procedure (Table I) can be regarded as only approximate, the calculated water requirement of 1·9 per cent by weight to cover the surface with a monolayer compares well with the 1·8 per cent required experimentally to reduce the alumina to minimum polarity (Activity " C "), and it would appear that the point of minimum polarity is reached when the surface of the alumina has been covered with a monolayer of water. No explanation can be offered as to why the polarity should increase with further additions of water.

TABLE I

Water Requirement of Alumina at Activity " B " to Cover Surface with a Monolayer

Surface area of alumina after evacuation at room temperature, m^2 per g	117·6
Water lost during evacuation, % w.w.	1·4
Surface area ≡ water lost,* m^2 per g	50·6
Available surface area, m^2 per g	67·0
Water ≡ 67·0 m^2 per g, % w.w.	1·9

* Mean area of water assumed 10·8 $Å^2$ per mol.

TABLE II

Retention Volume Results for Aluminas at Activity Levels " A " and " D "

Hydrocarbon	$U_g°$ value			
	Ethane	Propane	*n*-Butane	*n*-Pentane
Activity " A "	1·85	8·50	38·5	176·0
Activity " D "	0·575	1·70	4·90	14·0
Reduction factor	3·2	5·0	7·9	12·6

A feature of importance from the analytical point of view is that as the activity of the alumina decreases, so does the slope of the ethane–propane plots.

This means that considerable comparative reductions in the retention volumes of the higher boiling components can be obtained by the use of low activity aluminas. Table II shows the reductions in the retention volumes for ethane, propane, *n*-butane, and *n*-pentane (the figures for the latter two gases are extrapolated values) when the activity of the starting material (Activity " A ") is reduced to Activity " D."

RESULTS AND DISCUSSION (Fig 3)

Fig 3 shows the $U_g°$ values obtained when the alumina was subjected for one hour to treatment temperatures ranging from 200° to 1000° C. Because most of the elution peaks obtained with these aluminas were asymmetrical and retention volumes were extremely dependent on sample size and sample composition the $U_g°$ values can be regarded as only approximately relative values.

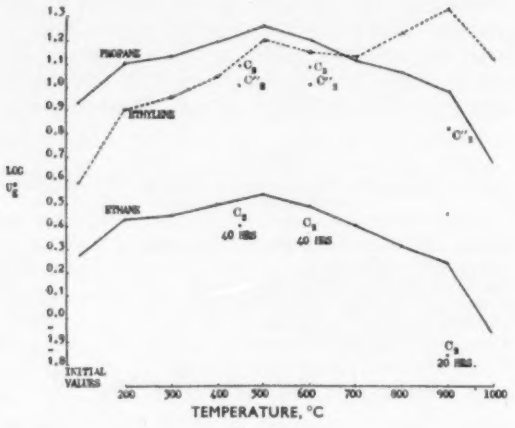

FIG 3

DATA ON ALUMINA HEAT-TREATED FOR ONE HOUR

The results obtained for treatment temperatures up to 500° C were mentioned in the previous section. Above 500° C, the $U_g°$ values for ethane and propane decrease. Carruthers and Gill [4] have shown that the surface areas of alumina hydrates decrease as calcination temperature increases above 500°–600° C. The dependence of the $U_g°$ values of the saturated hydrocarbons on the surface area of the alumina is therefore indicated. Evidence of some γ- to α-alumina phase transformation, which starts at about 1000° C, is shown by the sharp drop in $U_g°$ values obtained for the alumina heated at this temperature.

The values for ethylene decrease slightly with the first changes in surface area between 500° and 700° C, but rise again quite steeply at treatment temperatures of 800° and 900° C. At 1000° C, although there is a drop in the actual $U_g°$ value, there is still an increase in the polarity.

Table III shows the polarity as given by the ratio of the ethylene to propane values for the aluminas heat treated at temperatures from 200° to 1000° C.

TABLE III

Treatment Temperature v. *Polarity*

Treatment temp, ° C	Log $U_g°$ Ethylene (a)	Log $U_g°$ Propane (b)	Polarity as given by $\frac{(a)}{(b)}$
200 (1 hr)	0·90	1·10	0·82
300 (1 hr)	0·95	1·13	0·84
400 (1 hr)	1·04	1·19	0·87
500 (1 hr)	1·20	1·26	0·95
600 (1 hr)	1·15	1·20	0·96
700 (1 hr)	1·125	1·11	1·01
800 (1 hr)	1·23	1·055	1·17
900 (1 hr)	1·31	0·975	1·34
1000 (1 hr)	1·115	0·66	1·69
900 (16 hr)	0·81	0·45	1·80

From these results it is evident that the degree of polarity of an alumina is not governed by the resultant surface area of the alumina after heat treatment.

From a study of oxygen exchange between water and silica–alumina, Mills and Hindin [5] suggest that the removal of water during calcination leaves the surface in a strained and highly reactive condition. Cornelius *et al*,[3] discussing the high heats of adsorption of dehydrated aluminas, refer to the work of Weyl,[6] who studied the energy of solid surfaces when subjected to a high degree of dehydration and concluded that the high energy of such surfaces was due to strain derived from the unusual crystallographic distances involved in splitting off the water. Holm and Blue [7] studied the hydrogen–deuterium exchange activity of alumina and silica–alumina catalysts and found that pretreatment of catalysts at 650° C and 800° C instead of 400° C considerably increased the activity. Low activity was obtained when a preheated catalyst was exposed for a short time to a humid atmosphere, from which it was concluded that removal of traces of moisture held at a small number of highly active catalyst sites was responsible for the high activities obtained at high treatment temperatures. Such high activity sites are also the first to be poisoned by traces of moisture.

It thus seems reasonable to suggest that the polarity of an alumina, as indicated by the ratio of the logarithm of the $U_g°$ value of ethylene to propane, is subject to and indicative of the intensity of the high energies developed in the strained surface of the alumina when the last traces of water are split off. This is supported by the fact that exposure of a highly active alumina to a humid atmosphere for as short a period as 5 minutes can reduce the polarity value to less than 0·5. The reduction in the $U_g°$ value for ethylene when the alumina has been heated to 1000° C shows that the ethylene value, too, is controlled to some extent by the surface area of the alumina.

Also shown in Fig 3 are the results obtained by extending the period of heat treatment at 450°, 600°, and 900° C. After 40 hours at 450° and 600° C there are slight reductions in all values and it is evident, therefore, that in these temperature regions there is little point in continuing activation periods beyond one hour. Russell and Cochran [8] also found that there were no great differences in surface areas and weight losses between 1-hour and 16-hour activation periods in an investigation which covered this temperature region. At 900° C the drop in all values is quite considerable, but the polarity increases from 1·34 after one hour of treatment to 1·80 after 20 hours.

APPLICATION OF RESULTS TO SEPARATION OF HYDROGEN–HYDROCARBON GAS MIXTURES

The extent of the reductions in the retention volumes of the higher boiling hydrocarbons when low activity aluminas are used is such that the need to apply heat to the column to elute butenes and *iso*- and *n*-pentane is obviated. Fig 4 shows the separation effected on a gas sample using a column packing inactivated with water to give a $U_g°$ value of 0·60 for ethane.

Butene-1 and *iso*butene, which are not separated on partition columns (unless low temperatures or extremely long columns are used), are separated on

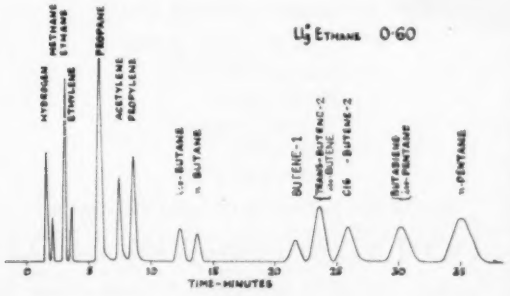

FIG 4

HYDROGEN–HYDROCARBON SEPARATION ON ALUMINA (100–150 MESH) INACTIVATED WITH WATER

Column: 8 ft × 6 mm i.d., 85 g alumina 15° C
Carrier gas: Nitrogen, 36 ml minute (corrected)

the inactivated alumina column. The *iso*butene peak is, however, coincident with the *trans*-butene-2 peak. Butadiene and *iso*pentane are also eluted together; their separation can, however, be achieved if a slight partitioning effect is introduced by replacing some of the water by another liquid. The separation obtainable when 2 per cent by weight of silicone oil is used to replace some of the water is shown in Fig 5. The methods for preparing the packings are given in Appendix I. Relative retention volume data are given in Appendix II. Because the polarity varies only very slightly with activity level in the region $U_g°$ ethane 0·45 to 0·8, the relative retention volume data given are approximate values to cover activities in this range.

For routine use it was found necessary to pre-saturate the carrier gas with water vapour to prevent

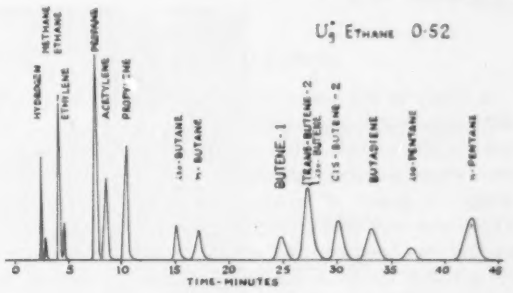

FIG 5

HYDROGEN–HYDROCARBON SEPARATION ON ALUMINA (100–150 MESH) INACTIVATED WITH 2% W.W. SILICONE OIL PLUS WATER

Column: 10 ft × 4 mm i.d., 54 g alumina 17° C
Carrier gas: Nitrogen, 16 ml minute (corrected)

it stripping water off the column. In practice, it was found that retention volumes for a given column remained constant for as long as 400 to 500 hours continuous use and then started to decrease (although order of elution remained the same). In such cases the pre-saturator was by-passed until sufficient water was driven off to return the retention volumes to their original value.

Carbon dioxide, hydrogen sulphide, and mercaptans are removed from the gas sample before injection on to the column by passing through a potash scrubber. Oxygen has the same retention time as hydrogen, but with the detector system used (hydrogen flame with nitrogen as carrier gas) it was found that up to 10 per cent by volume of air in a 1-ml gas sample caused no disturbance to the base-line, and any contamination of the sample with air during the scrubbing procedure did not affect the hydrogen response of the detector.

Attempts were made to reproduce the separations obtained with the water-inactivated column packings by using aluminas heated to temperatures in excess of 600° C and inactivated to the desired low level by the addition of a non-volatile liquid. Columns so prepared could be used without need to pre-saturate the carrier gas. However, due to partitioning of the hydrocarbon gases in the non-volatile liquids used (silicone oil, diglycerol, polyethylene glycol, and dimethyl sulpholane) the deactivation plots (as Fig 2 but for additions of non-volatile liquid instead of water) showed that there was no level of minimum or approximately constant polarity for an ethylene retention value intermediate between ethane and

propane retention values. Although it was found possible to obtain a combination of adsorptive and partition effects such that hydrogen, methane, and the other hydrocarbons could be separated, the affinity of the heated alumina for water vapour made the reproducible preparation of columns having the desired activity and polarity characteristics a matter of some difficulty.

CONCLUSIONS

A study of the adsorptive properties of alumina at different levels of activity, in terms of retention volumes for ethane, ethylene, and propane, has shown that when alumina is deactivated by the addition of water, a point of minimum polarity is reached. There is evidence that this point is reached when the surface has been covered with a monolayer of water. At activity levels in the proximity of this minimum polarity, the alumina makes a useful and easily prepared column packing for the separation, at room temperature, of refinery hydrocarbon gas mixtures containing hydrogen. A disadvantage of such columns is the need to pre-saturate the carrier gas with water vapour.

The activity of the aluminas, as shown by the retention volumes for ethane and propane, follows the trend of surface area. The polarity, as shown by the value for the unsaturated ethylene relative to propane, would appear to be indicative of the surface strain energy of the alumina. Although the gas chromatographic results obtained for activated aluminas cannot be regarded as absolute values because of their dependence on such variables as sample size and composition, relative values obtained under standard conditions could be used to characterize catalyst activity and polarity.

ACKNOWLEDGMENTS

Thanks are due to the management of Lobitos Oilfields Ltd for permission to publish this paper and to J. A. Lewis of Peter Spence and Sons Ltd, for his interest in the work and for providing the surface area measurement reported in Table I.

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APPENDIX I

PREPARATION OF COLUMN PACKINGS FOR THE SEPARATION OF HYDROGEN–HYDROCARBON GAS MIXTURES

Materials

B.D.H. "Chromatographic Grade" Alumina is sieved to give a 100–150 B.S. mesh fraction, which is stored in a tightly stoppered flask.

Gas chromatographic grade silicone oil.

Preparation of Water-deactivated Packing

Determine the $U_g°$ value for ethane using the sieved alumina in a 4 ft × 4 mm i.d. column.

Weigh 80 g of the alumina into a stoppered flask and add sufficient water to deactivate it to a $U_g°$ value of approximately 0·5 (the percentage of water required is shown below). Stopper the flask, shake well, and allow to stand for several hours, giving the flask an occasional shake.

Fill a 10-ft or 12-ft column (4 mm i.d.) by pouring in the deactivated alumina through a funnel while at the same time tapping or vibrating the sides of the column.

Preparation of Water–Silicone Oil Deactivated Packing

Add 1·4–1·6 g of silicone oil to 80 g alumina in a 250-ml beaker and mix with a spatula.

Transfer to a stoppered flask, determine the $U_g°$ value for ethane and add the amount of water required to deactivate to a $U_g°$ value of 0·5 for ethane.

Water Required to Deactivate 100–150 B.S. Mesh B.D.H. Alumina to $U_g°$ Value of 0·5 for Ethane

$U_g°$ Ethane	Approx w.w.% water
1·5	3·5
1·0	3·0
0·8	2·0
0·6	1·0

APPENDIX II

RELATIVE RETENTION VOLUME DATA

	B.D.H. alumina inactivated with water	B.D.H. alumina + 2% w.w. silicone oil + water
Methane	0·03	0·03
Ethane	0·12	0·12
Ethylene	0·15	0·15
Propane	0·35	0·35
Acetylene	0·45	0·40
Propylene	0·58	0·55
*iso*Butane	0·87	0·87
n-Butane	1·00	1·00
Butene-1	1·60	1·50
*iso*Butene, *trans*-Butene-2	1·80	1·70
cis-Butene-2	2·00	1·90
Butadiene	2·40	2·05
*iso*Pentane	2·40	2·35
n-Pentane	2·80	2·75

The above values are based on measurements from the hydrogen peak.

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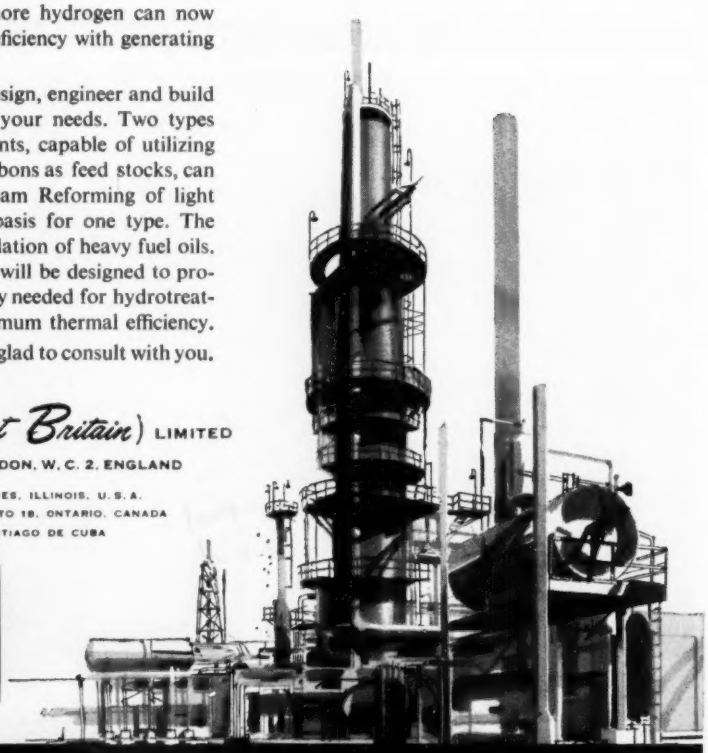

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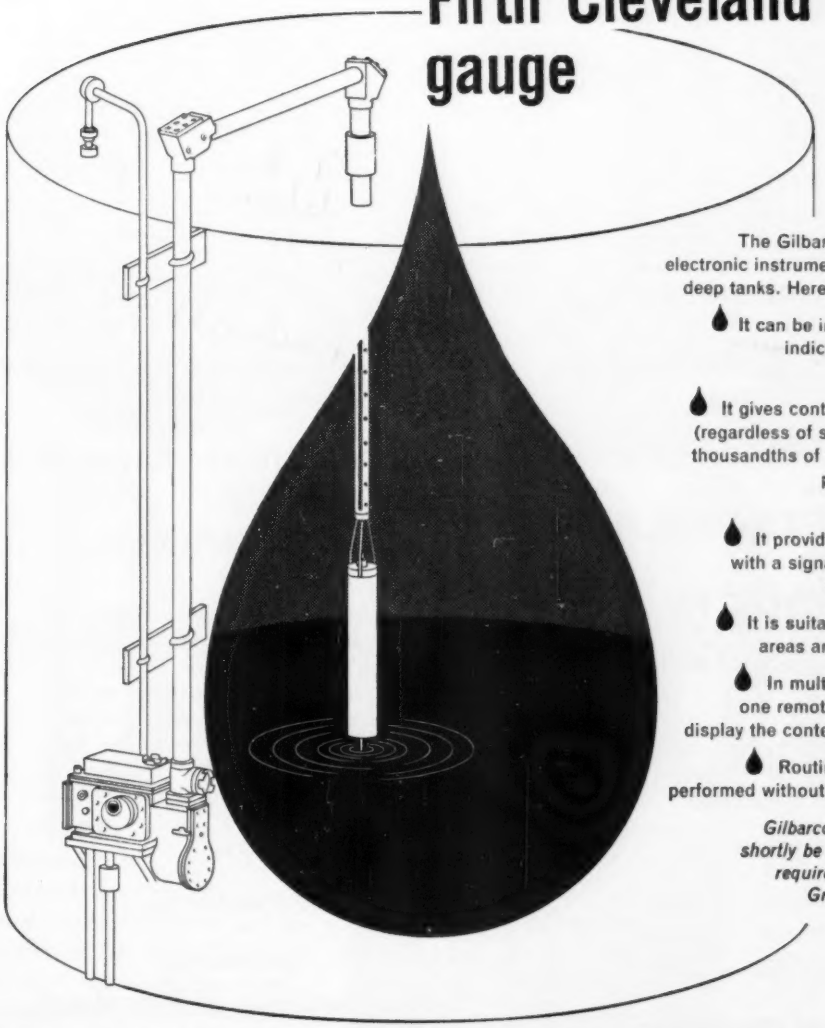

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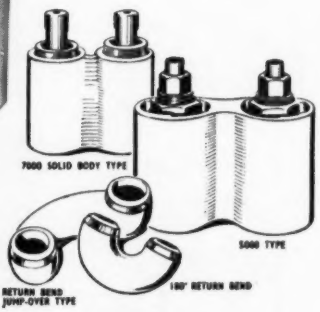

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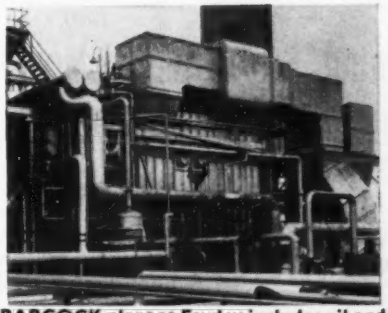

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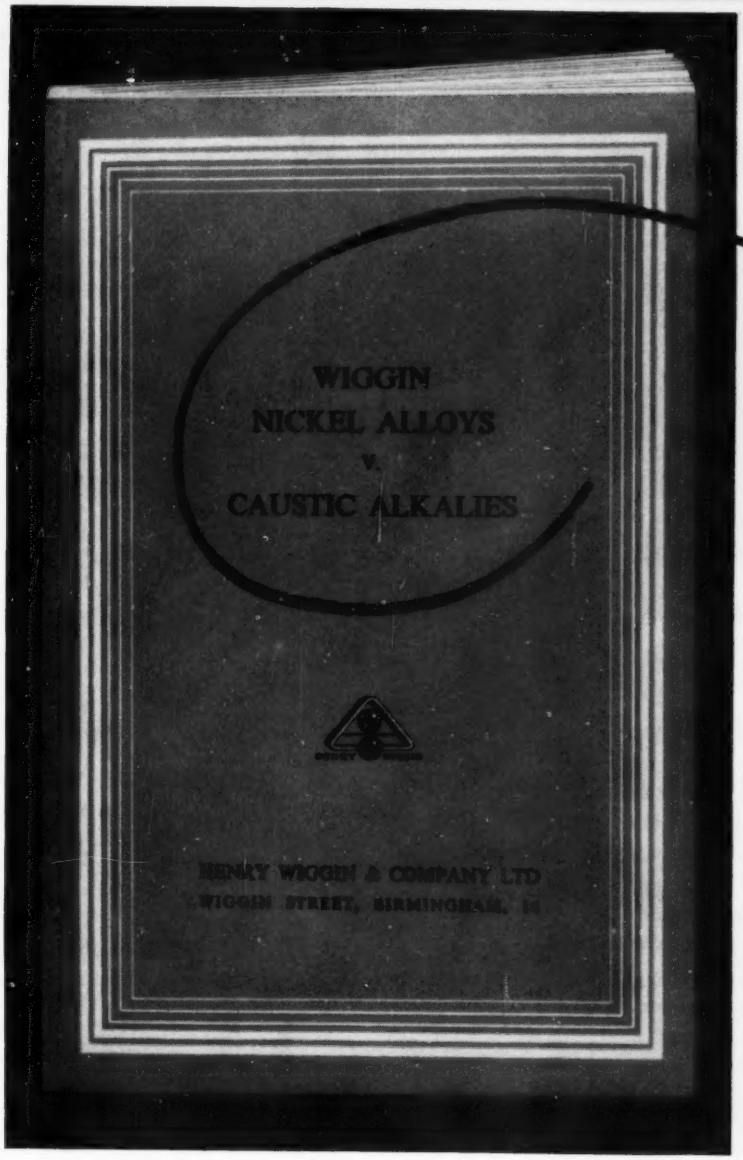

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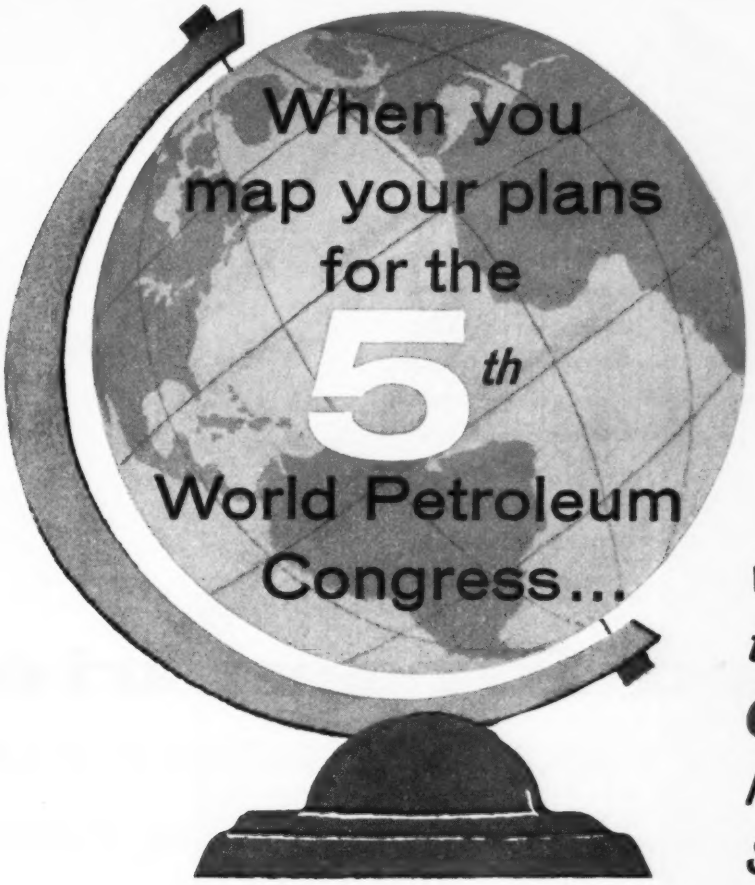

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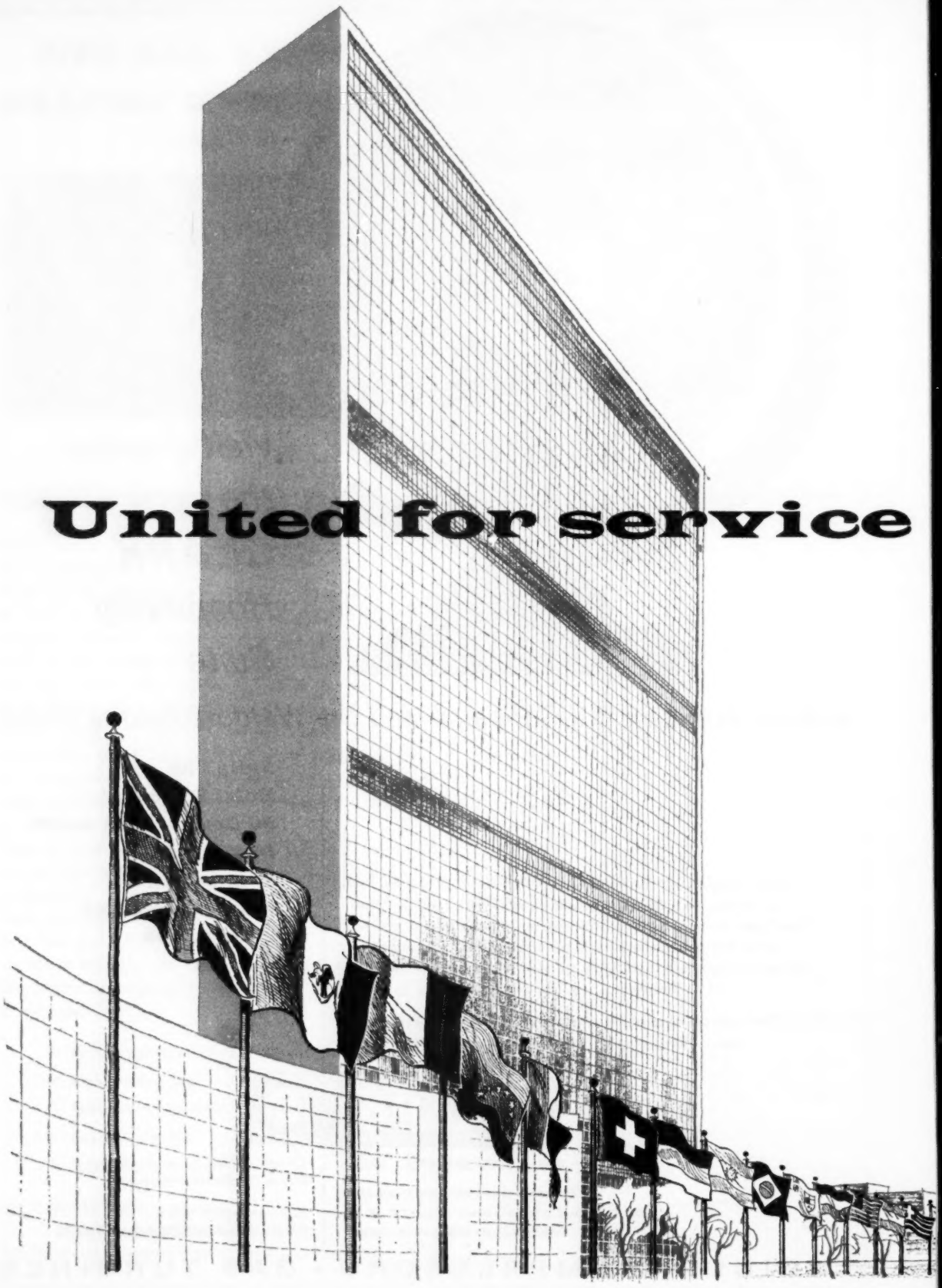

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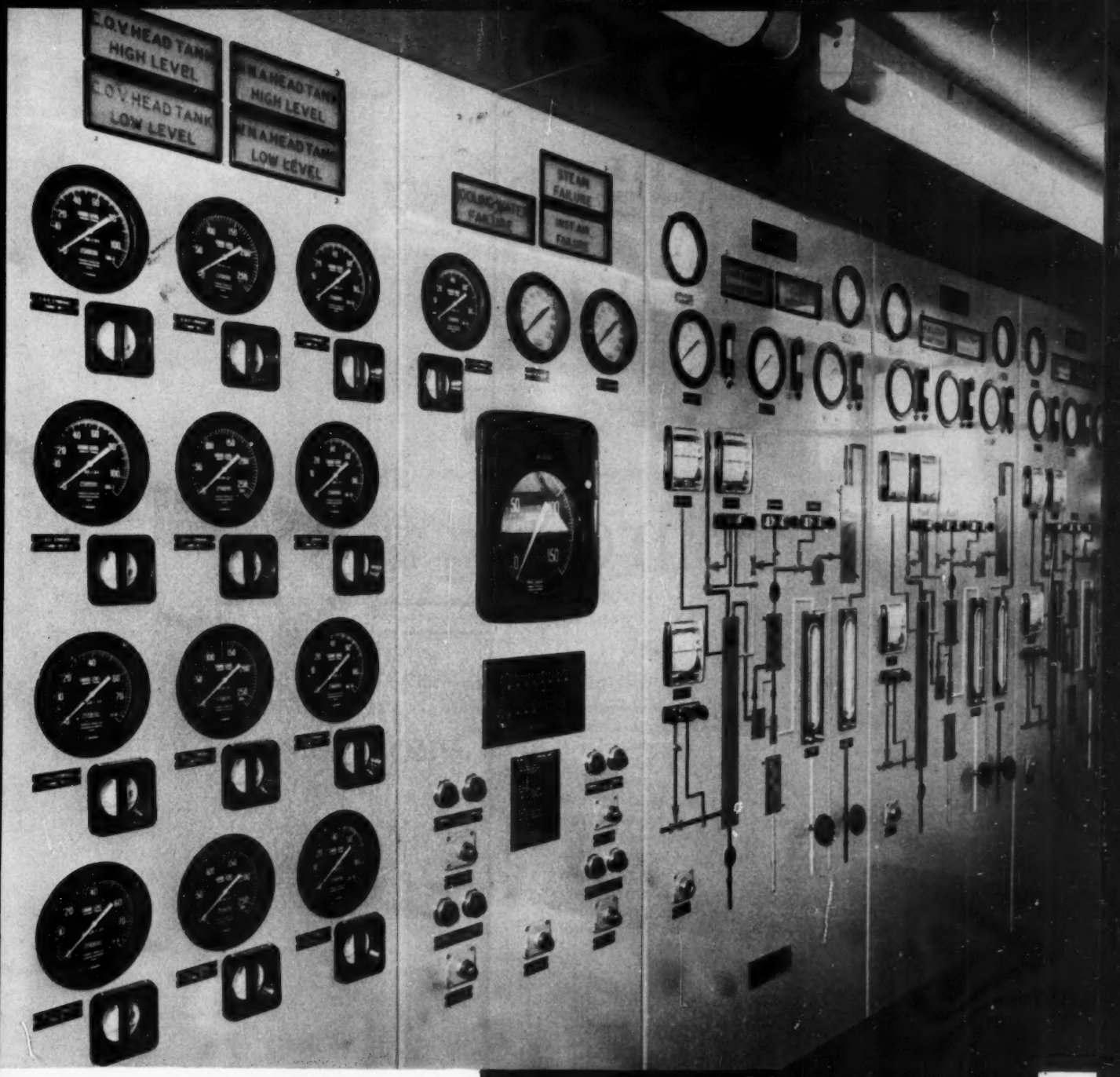

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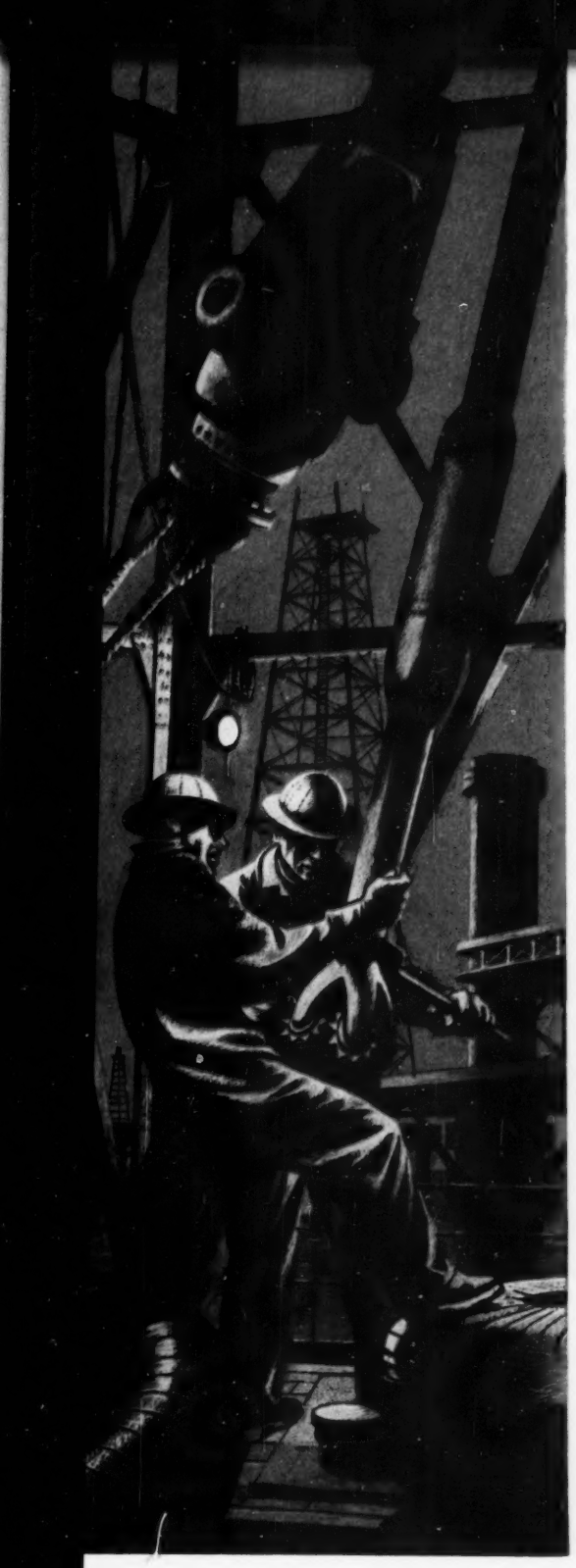

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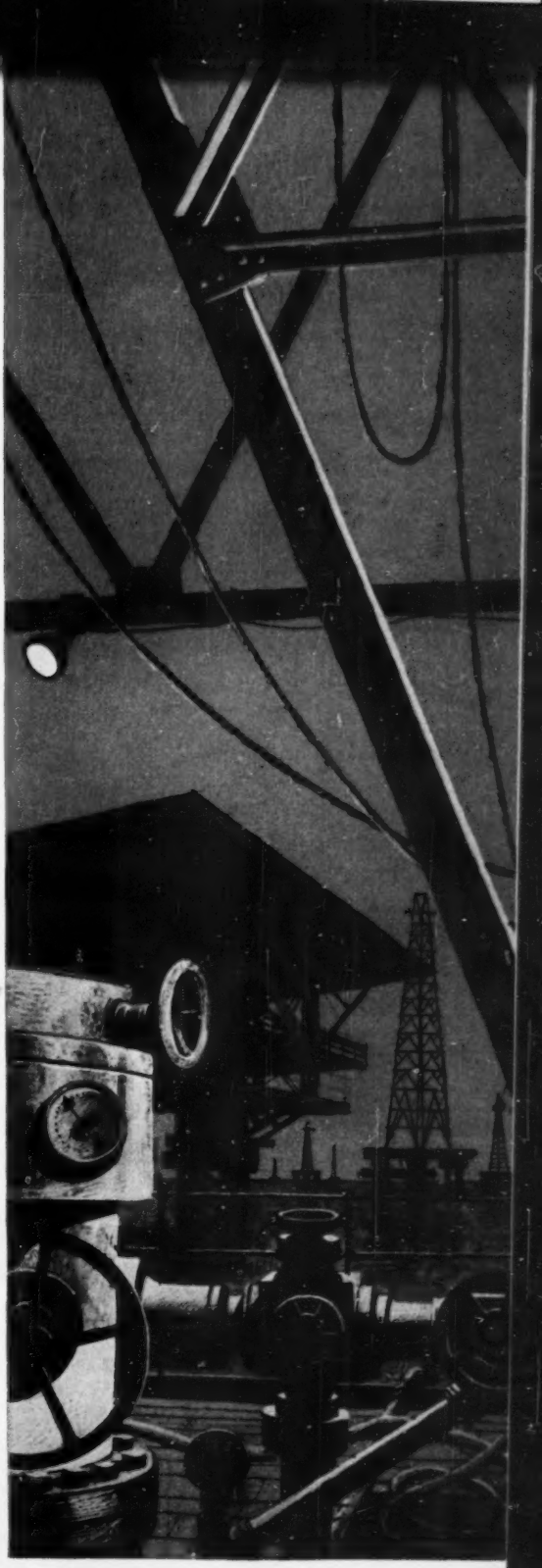

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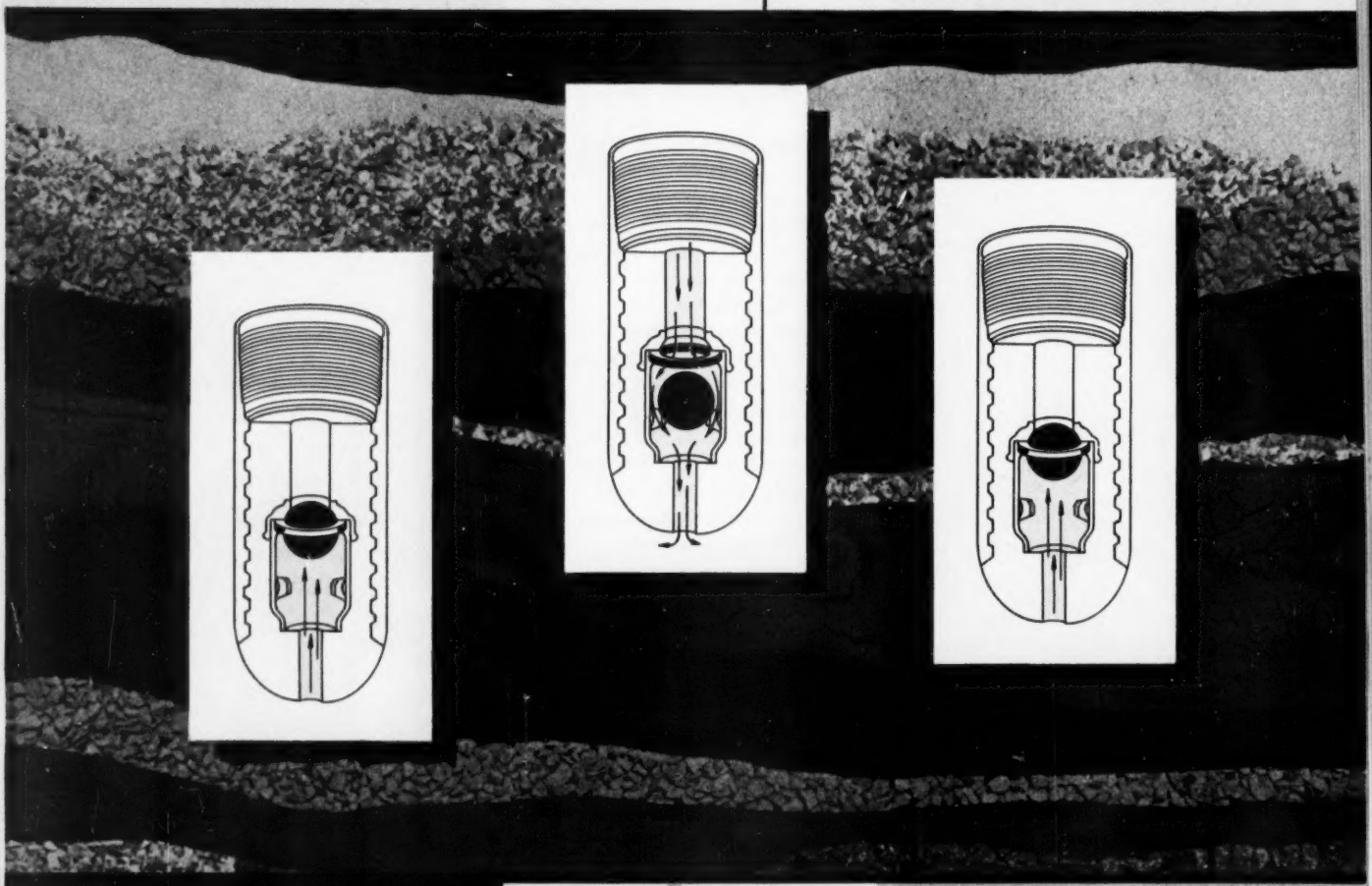

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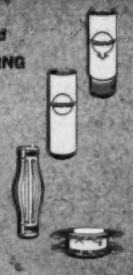

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in check

To check or contain an attack is part of the defensive strategy of both chess and refractories. Of refractories, or furnace linings, one may say they are always at the receiving end. Their job is to resist; to go on checking heat and the action of metals and slags, to ward off chemical erosion and withstand the increasingly severe effects of intensive furnace operations. It's life that counts in refractories. The new high quality products developed by General Refractories bring substantially increased life and new economies for all types of furnaces. Your best opening gambit is to consult the GR Technical Service Department. You will obtain authoritative advice on the correct selection and application of refractories for any specific problem.

Everything in Refractories

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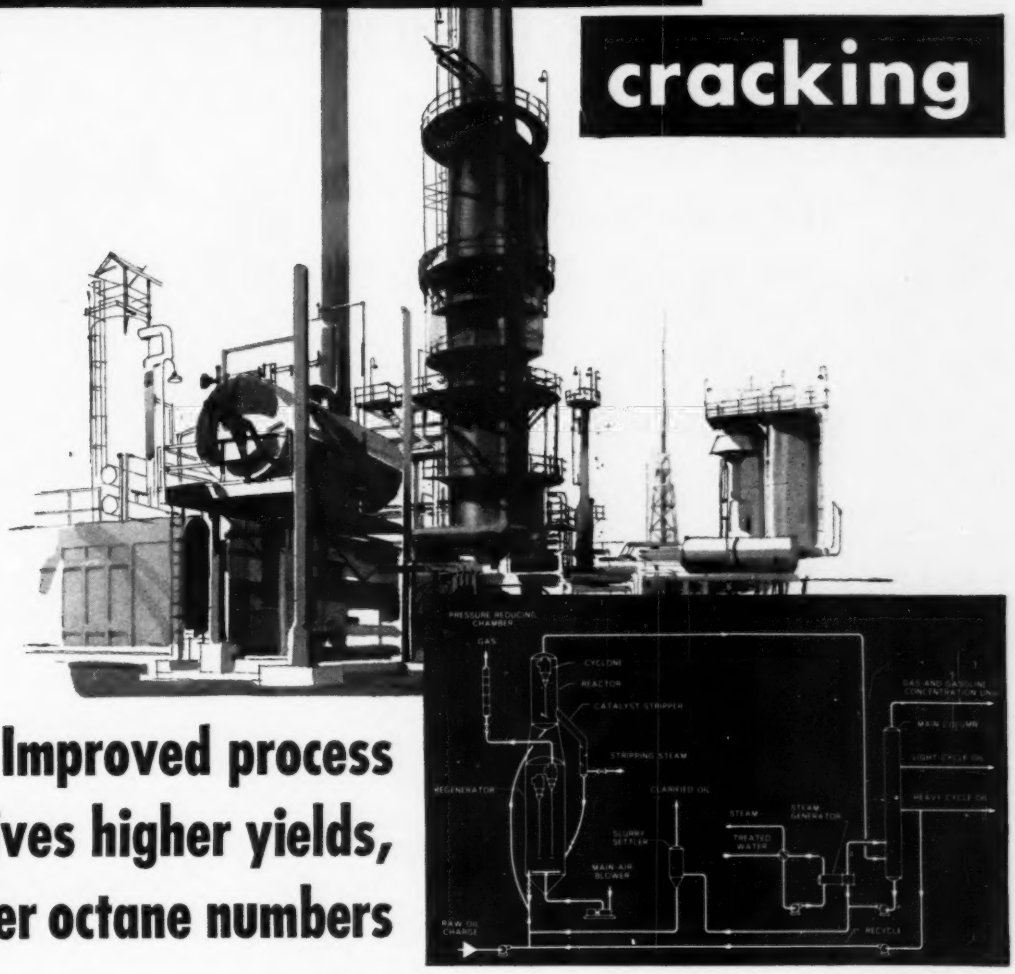
410

UOP fluid catalytic cracking



Improved process gives higher yields, higher octane numbers

An improved version of the basic and fluid catalytic cracking processes, UOP Fluid Catalytic Cracking converts low-value petroleum fractions to more valuable products. It produces gasoline, distillate fuels and gas for polymerization, alkylation, etc. under a better controlled, consequently more economical operation.

A less complicated, more easily operated, and efficient process, UOP Fluid Catalytic Cracking offers many advantages. The process requires lower original, operating and maintenance costs. UOP Fluid Catalytic Cracking gives longer on-stream time with minimum down-time. Catalyst inventory and consumption rates are low. UOP Fluid Catalytic Cracking can process either light or heavy oils including unvaporized feed stock. Positive contact between catalyst and charge stock assures efficient reaction and operation. Reactor space velocity can be changed, and reactor temperature controlled over a wide range to provide maximum flexibility in adjusting for various feed stocks or product changes.

Widely applied to refinery operations, UOP Fluid Catalytic Cracking offers a convincing record of proved performance.

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30 Algonquin Road, Des Plaines, Illinois, U.S.A.

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ESSO PETROLEUM COMPANY

Petroleum Chemical Plant at Fawley

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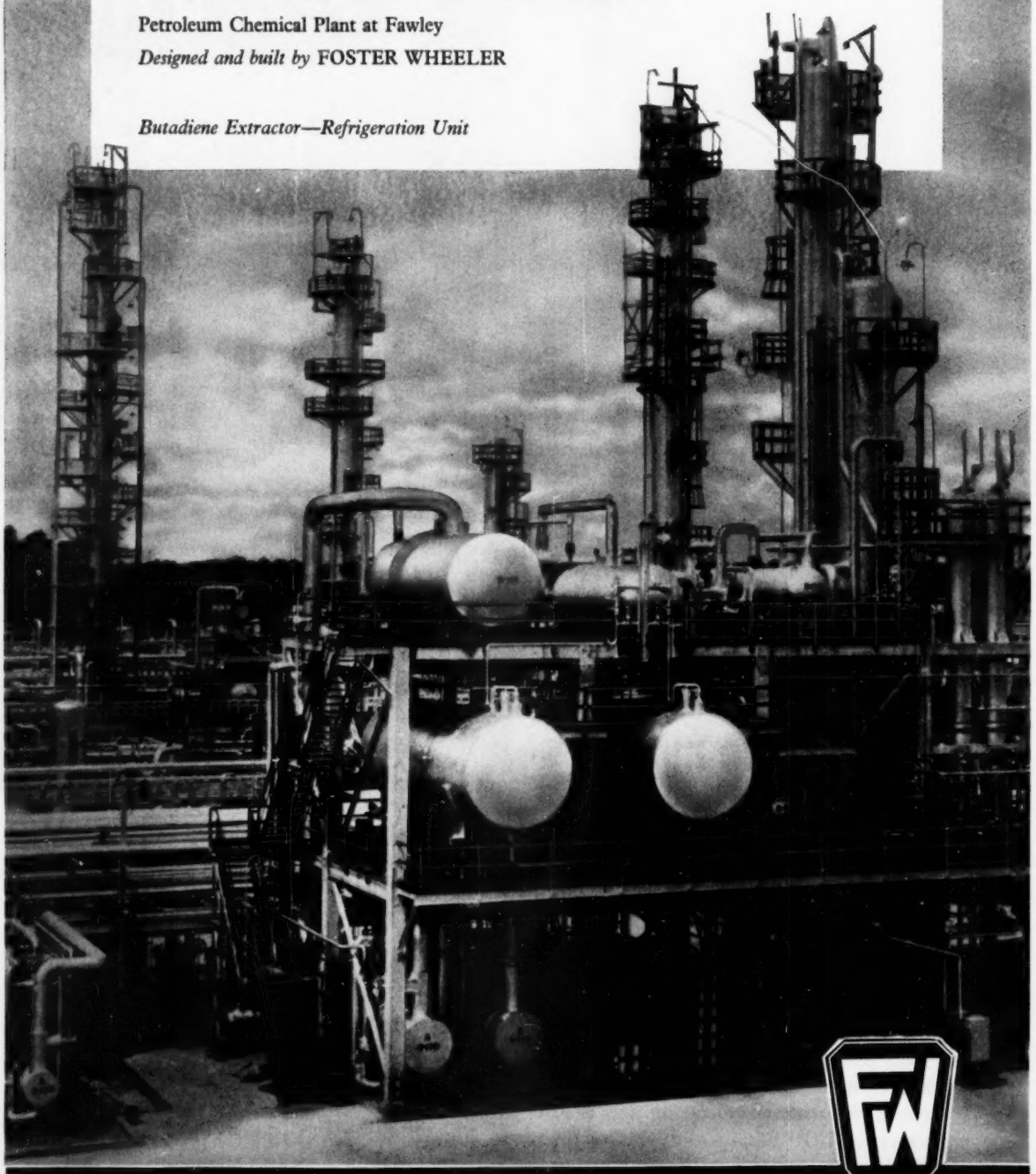
Butadiene Extractor—Refrigeration Unit



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